

EXHIBIT 23

PROCESS VALIDATION REPORT

BATCHES 4296A, 4300A, and 4301A

Revision No. 00

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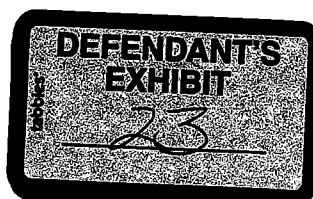
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Date: 11-18-74



AMIDE PHARMACEUTICAL, INC.

PROCESS VALIDATION SUMMARY

PRODUCT	DIGOXIN TABLETS, 0.5 mg	BATCH	4296A
			4300A
			4301A

The following comments apply to the three 400,000 tablet validation batches produced in this series.

This report includes data through Compression, which is the finished dosage form.

The process used to produce this batch follows exactly that shown in the normal batch record. Copies of the actual batch records are available in the file.

The data supporting the validation of the analytical methods used may be found in the Analytical Method Validation Report issued for this product.

A copy of the protocol to be followed for this project is included.

Evaluation of the data includes calculation of the Process Capability Index, Cp, when appropriate. Cp is a measure of the ability of a process to produce material that is all within the specification range. It verifies that the entire distribution curve for the data collected falls within the allowable limits. The following equation is used.

$$Cp = \frac{(\text{Upper Limit} - \text{Lower Limit})}{6 \times \text{St. Dev.}}$$

Any value equal to or greater than 1 is acceptable.

**AMIDE PHARMACEUTICAL, INC.
PROCESS VALIDATION**

DIGOXIN TABLETS, 0.5 mg MPR NO. 14702 - 00

CONCLUSIONS AND OBSERVATIONS

All samples met the established acceptance criteria.

Based on these three batches, the process is considered validated and is acceptable for use.

The data verifies the initial acceptance criteria for all parameters. At this point no revision to any of these ranges will be made.

The final blends showed adequate uniformity for all batches. The resulting Cp value is 2.1, which is more than acceptable.

Content uniformity results are all within the acceptance criteria, and are essentially comparable to the blend results.

Results for both the final blends and content uniformity center around the label amount.

All Dissolution samples for the three batches met the USP requirements. The values for the three batches are comparable, however there is some variability within the individual batches.

The data for each protocol step follows a summary of that step, in the order in which it appears in the protocol.

Amide Pharmaceutical, Inc.**Process Validation****DIGOXIN TABLETS, 0.5 mg****Process Validation Summary**

Test	Initial Limits	Batch	4296A	4300A	4301A	Combined	Final Limits
Final Blend Assay (%)	85.0 - 115.0 %Th. (Ind.)	Average	98.8	99.5	100.1	99.4	85.0 - 115.0 %Th. (Ind.)
		Std Dev	0.8	1.6	2.0	1.6	90.0 - 110.0 %Th. (Avg.)
		Cp				2.1	
Compression Weight (g)	0.123 - 0.137 g	Average	0.130	0.131	0.131	0.131	0.123 - 0.137 g
		Std Dev	0.002	0.002	0.001	0.002	
		Cp				1.5	
Compression Hardness (Kp)	2.0 - 8.0 kp	Average	4.3	4.9	4.9	4.7	2.0 - 8.0 kp
		Std Dev	0.4	0.3	0.4	0.5	
		Cp				2.0	
Compression Thickness (mm)	3.0 - 4.0 mm	Average	3.36	3.33	3.32	3.34	3.0 - 4.0 mm
		Std Dev	0.02	0.03	0.02	0.03	
		Cp				6.1	
Compression Friability (%)	NMT: 1.0 %	Average	0.04	0.05	0.04	0.04	NMT: 1.0 %
		Std Dev	0.01	0.01	0.01	0.01	
		Average	3.5	3.5	3.5	3.5	
Compression Disintegration (min)	N/A	Std Dev	0.5	0.5	0.5	0.5	N/A
Compression Content Uniformity (%)	85.0 - 115.0 % RSD NMT: 6.0 %	Average	101.8	100.9	99.8	100.8	85.0 - 115.0 % RSD NMT: 6.0 %
		Std Dev	2.8	1.4	1.6	2.2	
		Cp				2.3	
Compression Disolution (%) 15 min.	NMT: 90% (ind.)	Average	82.3	79.0	79.9	80.4	NMT: 90% (ind.)
		Std Dev	2.3	3.1	1.7	2.8	
Compression Disolution (%) 60 min.	NLT: 80% (avg)	Average	94.0	91.1	94.1	93.0	NLT: 80% (avg)
		Std Dev	6.4	3.4	4.3	5.0	

AMIDE PHARMACEUTICAL, INC.
PROCESS VALIDATION

DIGOXIN TABLETS, 0.5 mg MPR NO. 14702 - 00

PROTOCOL STEP - RAW MATERIALS

The raw materials used will be tested, as stated in the protocol, in accordance with approved specifications and methods. In addition, bulk density, tamped density and particle size distribution will be included.

ACCEPTANCE CRITERIA

Parameters normally evaluated will be compared to the current specifications. The density and particle size data will be gathered and used to formulate guidelines when sufficient data is accumulated.

RESULTS - See attached data summary sheets.

CONCLUSIONS AND COMMENTS

All data is acceptable.

Any differences noted do not appear to have any effect on finished product quality.

Particle size determinations were run on two different pieces of equipment. One is a "Ro-Tap" type unit and the other a Micron Air Jet Sieve. For samples run on the "Ro-Tap" the coarser mesh screen is listed first.

It should be noted that particle size and density evaluation was not done for the Green Lake Blend. Since this material is present in such a small amount any differences in either of these parameters will have no significant effect on the final blend.

AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg****Rau Material Usage Chart**

Item #	Item Name	Batch # 4296A P.O. #	Batch # 4300A P.O. #	Batch # 4301A P.O. #
3115	Corn Starch, NF	2961	2961	2961
0111	Digoxin, USP	2967 & 2629-1	2629-1	2629-1
3038	Green Lake Blend LB 603	3245	3245	3245
3000	Croscarmellose Sodium, NF	3799	3799	3799
3051	Lactose Hydrous Impalpable, NF	3855	3855	3855
3088	Starch Pregelatinized, NF	3789-1	3789-1	3789-1
3059	Microcrystalline Cellulose, NF	3909	3909	3909
3050	Lactose Anhydrous, NF	4015	4015	4015
3089	Stearic Acid, NF	3910	3910	3910
3081	Silicon Dioxide, NF	3696	3696	3696

AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg**

Raw Material Comparison - Corn Starch, NF (3115)

P.O. #	2961
Test Type	Initial
Manufacturer	National Starch & Chem
Manufacturer Lot #	MC-7621

PARAMETERS	SPECIFICATIONS	RESULTS
DESCRIPTION	Passes Test	Passes
IDENTIFICATION A	Positive	Passes
IDENTIFICATION B	Positive	Passes
MICROBIAL LIMITS	Passes Test	Passes
PH	4.5 - 7.0	4.9
LOSS ON DRYING	NMT 14.0%	0.8%
RESIDUE ON IGNITION	NMT 0.5%	0.1%
IRON	NMT 0.002%	< 0.002%
OXIDIZING SUBSTANCES	Passes Test	Passes
SULFUR DIOXIDE	Passes Test	Passes
BULK DENSITY		0.55 g/mL
TAP DENSITY		0.69 g/mL
PARTICAL SIZE (US 325)	% Retained	48.0%
PARTICAL SIZE (US 200)	% Retained	7.9%
PARTICAL SIZE (US 100)	% Retained	NIL

AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg**

Raw Material Comparison - Digoxin USP (0111)

P.O. #	2629-1	2967
Test Type	Initial	Initial
Manufacturer	Boehringer Ingelheim	Boehringer Ingelheim
Manufacturer Lot #	240180	240180

PARAMETERS	SPECIFICATIONS	RESULTS	RESULTS
DESCRIPTION	Fine white powder	Passes	Passes
IDENTIFICATION A	IR spectrum corr. to std.	Passes	Passes
IDENTIFICATION B	Retention time corr. to std.	Passes	Passes
IDENTIFICATION C	Rf value of blue spot corr. to std.	Passes	Passes
LOSS ON DRYING	NMT 1.0%	0.6%	0.6%
RESIDUE ON IGNITION	NMT 0.5%	0.1%	0.1%
RELATED GLYCOSIDES	NMT 3% as digitoxin	< 3%	< 3%
ASSAY	95.0 - 101.0%	98.6%	98.6%
BULK DENSITY		0.24 g/ml	0.24 g/ml
TAP DENSITY		0.37 g/ml	0.37 g/ml
PARTICLE SIZE (US 200)	%Retained	95.6%	95.6%
PARTICLE SIZE (US 325)	%Retained	4.0%	4.0%

AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg**

Raw Material Comparison - Green Lake Blend LB 603 (3038)

P.O. #	3245
Test Type	Initial
Manufacturer	Colorcon
Manufacturer Lot #	208181

DESCRIPTION	Green odorless fine powder.	Passes
IDENTIFICATION	Max absorption at 630 + or - 5 nm	Passes

AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION*****DIGOXIN TABLETS, 0.5 mg***

Raw Material Comparison - Croscarmellose Sodium, NF (3000)

P.O. #	3799
Test Type	Initial
Manufacturer	FTC
Manufacturer Lot #	T423N

PARAMETERS	SPECIFICATIONS	
DESCRIPTION	Passes Test	Passes
IDENTIFICATION A	Positive	Passes
IDENTIFICATION B	Positive	Passes
IDENTIFICATION C	Positive	Passes
pH	5.0 - 7.0	6.3
LOSS ON DRYING	NMT 10.0%	2.3%
HEAVY METALS	NMT 0.001%	< 0.001%
SODIUM CHLORIDE & SODIUM STARCH GLYCOLATE	NMT 0.5%	0.21%
DEGREE OF SUBSTITUTION	0.60 to 0.85	0.7
CONTENT OF WATER SOLUBLE MATERIAL	1.0% - 10.0%	3.7%
SETTLING VOLUME	10.0 mL - 30.0 mL	23 mL
MICROBIAL TEST		Passes
BULK DENSITY		0.50 g/mL
TAP DENSITY		0.72 g/mL
PARTICLE SIZE (US 325)	% Retained	0.8%
PARTICLE SIZE (US 200)	% Retained	0.4%

AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg**

Raw Material Comparison - Lactose Hydrous Impalpable NF (3051)

P.O. #	3855
Test Type	Initial
Manufacturer	HMS Chemical
Manufacturer Lot #	16478

PARAMETERS	SPECIFICATIONS	RESULTS
DESCRIPTION	Passes Test	Passes
IDENTIFICATION A	Positive	Passes
IDENTIFICATION B	Positive	Passes
CLARITY AND COLOR OF SOLUTION	Passes Test	Passes
LOSS ON DRYING	NMT 1.0%	0.1%
SPECIFIC ROTATION	+54.8° to +55.5°	+55.3°
MICROBIAL LIMITS	Passes Test	Passes
WATER	Hydrous: NMT 5.5%	5.0%
RESIDUE ON IGNITION	NMT 0.1%	0.03%
HEAVY METALS	NMT 5 ppm	< 5 ppm
ACIDITY/ALKALINITY	Passes Test	Passes
PROTEIN/LIGHT ABSORBING IMPUR.	Passes Test	Passes
ORGANIC VOLATILE IMPURITIES	Passes Test	Passes
BULK DENSITY		0.58 g/mL
TAP DENSITY		0.89 g/mL
PARTICAL SIZE (US 325)	% Retained	82.8%
PARTICAL SIZE (US 200)	% Retained	11.6%

AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg**

Raw Material Comparison - Starch Pregelatinized NF (3088)

P.O. #	3789-1
Test Type	Initial
Manufacturer	Colorcon
Manufacturer Lot #	407056

PARAMETERS	SPECIFICATIONS	RESULTS
DESCRIPTION	Passes Test	Passes
IDENTIFICATION	Positive	Passes
PH	4.5 - 7.0	6.1
IRON	NMT 0.002%	<0.002 %
OXIDIZING SUBSTANCES	Passes Test	Passes
SULFUR DIOXIDE	NMT: 0.008%	Passes
MICROBIAL LIMITS	Passes Test	Passes
LOSS ON DRYING	NMT 14.0%	9.4 %
RESIDUE ON IGNITION	NMT 0.5%	0.2%
BULK DENSITY		0.65 g/mL
TAP DENSITY		0.83 g/mL
PARTICAL SIZE (US 100)	% Accumulation	2.8 %
PARTICAL SIZE (US 200)	% Accumulation	24.2 %
PARTICAL SIZE (US 325)	% Accumulation	47.2 %

AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg**

Rau Material Comparison - Microcrystalline Cellulose, NF (3059)

P.O. #	3909
Test Type	Initial
Manufacturer	Mendell
Manufacturer Lot #	4577

PARAMETERS	SPECIFICATIONS	RESULTS
DESCRIPTION	Passes Test	Passes
IDENTIFICATION	Positive	Passes
PH	5.5 to 7.0	6.5
LOSS ON DRYING	MT 5.0%	3.2%
RESIDUE ON IGNITION	MT 0.05%	0.01%
WATER SOLUBLE SUBSTANCES	MT 0.16%	0.10%
HEAVY METALS	MT 0.001%	<0.001%
STARCH	Passes Test	Passes
ASSAY	97.0% - 102.0%	98.8%
BULK DENSITY		0.32 g/m
TAP DENSITY		0.42 g/m
PARTICAL SIZE (US 325)	% Retained	40.1
PARTICAL SIZE (US 200)	% Retained	17.0
PARTICAL SIZE (US 100)	% Retained	N11

AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg**

Rau Material Comparison - Lactose Anhydrous, NF (DT) (3050)

P.O. #	4015
Test Type	Initial
Manufacturer	Quest
Manufacturer Lot #	MRP545363

PARAMETERS	SPECIFICATIONS	RESULTS
DESCRIPTION	Passes Test	Passes
IDENTIFICATION A	Positive	Passes
IDENTIFICATION B	Positive	Passes
CLARITY AND COLOR OF SOLUTION	Passes Test	Passes
LOSS ON DRYING	NMT 0.5%	0.2%
SPECIFIC ROTATION	Between +54.8° and +55.5°	+55.2°
MICROBIAL LIMITS	NMT 100 per gm	Passes
WATER	NMT 1.0%	0.4%
RESIDUE ON IGNITION	NMT 0.1%	0.04%
HEAVY METALS	NMT 5 ppm	< 5 ppm
ACIDITY/ALKALINITY	Passes Test	Passes
PROTEIN AND LIGHT ABSORBING IMPURITIES	NMT 0.25	Passes
ORGANIC VOLATILE IMPURITIES	Passes Test	Passes
BULK DENSITY		0.57 g/ml
TAP DENSITY		0.81 g/ml
PARTIAL SIZE (US 100)	% Accumulation	13.1%
PARTIAL SIZE (US 200)	% Accumulation	28.6%
PARTIAL SIZE (US 325)	% Accumulation	40.5%

AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg**

Raw Material Comparison - Stearic Acid, NF (3089)

P.O. #	3910
Test Type	Initial
Manufacturer	Witco
Manufacturer Lot #	440069

PARAMETERS	SPECIFICATIONS	RESULTS
DESCRIPTION	Passes Test	Passes
CONGEALING TEMPERATURE	NLT 54°	55°
RESIDUE ON IGNITION	NMT 0.1%	0.01
HEAVY METALS	NMT 0.001%	<0.001
MINERAL ACID	Passes Test	Passes
NEUTRAL FAT OR PARAFIN	Passes Test	Passes
IODINE VALUE	NMT 4	0.10
ASSAY A	NLT 40.0%	43.4%
ASSAY B	NLT 90.0%	96.4%
ORGANIC VOLATILE IMPURITIES	Passes Test	Passes
BULK DENSITY		0.38 g/ml
TAP DENSITY		0.49 g/ml
PARTICAL SIZE (US 325)	% Retained	54.0%
PARTICAL SIZE (US 200)	% Retained	6.4%

AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg**

Raw Material Comparison - Silicon Dioxide, NF (3081)

P.O. #	3696
Test Type	Initial
Manufacturer	Dequessa Corp.
Manufacturer Lot #	4-90

PARAMETERS	SPECIFICATIONS	RESULTS
DESCRIPTION	Passes Test	Passes
IDENTIFICATION	Positive	Passes
PH	4 - 8	6.7
LOSS ON DRYING	NMT 5.0%	4.0%
CHLORIDE	NMT 0.1%	<0.1%
SULFATE	NMT 0.5%	<0.5%
ARSENIC	NMT 3 ppm	<3 ppm
HEAVY METAL	NMT 0.003%	<0.003%
ASSAY	NLT 99.0%	99.6%
BULK DENSITY		0.10 g/ml
TAP DENSITY		0.13 g/ml
PARTICAL SIZE (US 325)	% Retained	N11

AMIDE PHARMACEUTICAL, INC.
PROCESS VALIDATION

DIGOXIN TABLETS, 0.5 mg MPR NO. 14702 - 00

PROTOCOL STEP - TEMPERATURE/HUMIDITY READINGS

A temperature and humidity reading will be taken once each day in the production area. The production dates for the three batches are as follows:

4296A	10/14/94 - 10/18/94
4300A	10/18/94 - 10/21/94
4301A	10/18/94 - 10/21/94

RESULTS - See attached data summary sheets.

CONCLUSIONS AND COMMENTS

The temperature ranged from 69 - 72 degrees F, and the relative humidity from 48 - 67 %. This indicates that acceptable product can be made at these levels.

AMIDE PHARMACEUTICAL, INC.

TEMPERATURE/HUMIDITY READINGS

PERIOD COVERING DIGOXIN TABLETS, 0.5 mg

BATCHES 4296A, 4300A, AND 4301A

LOCATION	DATE	TEMP. (Deg. F)	RH (%)
Near Pr. Rm. #1	14-Oct-94	70	54
Near Pr. Rm. #1	17-Oct-94	69	48
Near Pr. Rm. #1	18-Oct-94	70	56
Near Pr. Rm. #1	19-Oct-94	71	58
Near Pr. Rm. #1	20-Oct-94	72	65
Near Pr. Rm. #1	21-Oct-94	72	67

AMIDE PHARMACEUTICAL, INC.
PROCESS VALIDATION

DIGOXIN TABLETS, 0.5 mg MPR NO. 14702 - 00

PROTOCOL STEP - BLEND UNIFORMITY

Utilizing a sampling thief, sample each of the blenders from the positions shown on the attached data summary. Separately analyze, and report, each one for active ingredient content.

The speed of each blender will be monitored both empty and at each stage of blending.

ACCEPTANCE CRITERIA

Final Blend - 85.0 - 115.0 % Th. (Individual)

RESULTS - See the attached data summary.

CONCLUSIONS AND COMMENTS

The final blends for the three batches met all acceptance criteria and appear to be uniformly blended.

The bulk and tamped density results are comparable for all three batches.

Some differences are evident in the particle size distribution for the three batches. This is especially noticeable for the 100 and 200 mesh screens. Batches 4300A, and 4301A, are comparable to one another, while batch 4296A is significantly finer. These differences do not correlate with either the content uniformity or dissolution results. Therefore this is considered normal variation.

The speed for both blenders was observed to be constant throughout production of the three batches. The same speed was obtained both empty and under load. The supporting documentation is attached.

1 Cu. Ft. (31) - 26 rpm

3 Cu. Ft. (32) - 22 rpm

AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg**

Final Blend - Assay (% Label)

Batch	4296A	4300A	4301A
Left Column - Top Left	97.7	98.6	98.7
Left Column - Top Center	98.4	100.2	103.1
Left Column - Top Right	98.6	99.2	100.9
Right Column - Top Left	97.6	96.2	95.9
Right Column - Top Center	98.9	98.9	99.9
Right Column - Top Right	99.4	98.3	99.3
Middle Left	98.3	99.4	101.2
Middle Center	99.7	99.6	100.2
Middle Right	99.9	102.5	102.8
Bottom Left	99.2	100.2	98.7
Bottom Right	98.7	101.0	100.3
Average	98.8	99.5	100.1
St Dev.	0.8	1.6	2.0
RSD	0.8	1.6	2.0

AMIDE PHARMACEUTICAL, INC.

PROCESS VALIDATION

DIGOXIN TABLETS, 0.5 mg

Final Blend – Density/Particle Size

Density (g/ml)

Batch	4296A	4300A	4301A
Bulk	0.62	0.61	0.61
Tap	0.91	0.87	0.87

Particle Size (% Retained)

Batch	4296A	4300A	4301A
325	43.4	47.2	52.9
200	15.3	34.1	32.9
100	1.9	14.2	13.9
60	N11	3.2	3.5
40	N/A	N11	N11

PRODUCT NAME (1): Digoxin TCB 0.5mg (147)

BATCH #: 4296A

MPR #: 14702

REV # : 00

DATE: 10/14/94

BLENDER #: 31

PD2-046

Je Pharmaceutical, Inc.

PROCESS VALIDATION DATA SHEETPRODUCT NAME (#): Digoxin Tablets 0.5mg (147)BATCH #: 4296AMPR #: 14702REV #: 00DATE: 10/14/94BLENDER #: 32

TIME	BLENDER'S CONTENTS	BLENDER'S THEO. MATERIAL WEIGHT (Kg)	BLENDER'S RPM	DONE BY	CHECKED BY
4:10 PM	^{J.D. 10/14/94} EMPTY EMPTY	0	22	KI	J.D.
5:03 PM	Corn starch, Digoxin, Greenlake Croscarmellose, Lactose Anhydrous		22	KI	J.D.
	Starch, microcrystalline	33.80 Kg	33-80 ^{J.D. 10/14/94}		
5:25 PM	Corn starch, Digoxin, Greenlake Croscarmellose, Lactose Anhydrous		22		
	Starch, microcrystalline		^{J.D.} 10/14/94		
	Lactose Anhydrous	50.80 Kg	50.80	KI	J.D.
	Corn starch, Digoxin, Greenlake Croscarmellose, Lactose Anhydrous				
	Starch, microcrystalline				
5:47 PM	Lactose Anhydrous, Stearic Acid Silicon Dioxide	52.00 Kg	22	J.D.	AN

PD2-046

PRODUCT NAME (#): Digoxin Tablets 0.5mg (147)

BLENDER #: 31

PD2-046

PRODUCT NAME (1): Digoxin Tablet 0.5mg (147)

MPR #: 14702

REV 1: 00

DATE: 10/18/94

BLENDER #: 32

PDZ-046

PRODUCT NAME (1): Digoxin Tablets 0.5mg (147)

MPR #: 14702REV 1: 00

DATE: 10/18/94

BLENDER #: 31

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ACTAV 001944344

PRODUCT NAME (1): Digoxin Tablets 0.5mg (147)

BLENDER #: 32

PD2-046

AMIDE PHARMACEUTICAL, INC.
PROCESS VALIDATION

DIGOXIN TABLETS, 0.5 mg MPR NO. 14702 - 00

PROTOCOL STEP - COMPRESSION

Samples were taken from each side of the press each 15 minutes and were evaluated for the following parameters.

Weight (n = 10)
Thickness (n = 5)
Hardness (n = 5)

These samples will be arranged chronologically and the batch divided into thirds. Front and rear will be analyzed separately as follows.

Friability	10 g - 1 run
Dissolution	N = 12 (6 front & 6 rear)
Disintegration	N = 6

Content uniformity is to be run across the entire batch. One tablet from each sample taken is to be run from the front, and one from the rear. A minimum of 30 is required from each side.

During compression a minimum quantity of tablets will be run at speeds higher and lower than normal. The actual speeds will be selected during production. These tablets will be evaluated for weight and hardness.

During compression minimum quantities of tablets will be run at hardness of 0.5 - 3 KP and greater than 8 KP. An attempt will also be made to run some tablets at the highest possible hardness that can be obtained without capping. These tablets will be evaluated for Dissolution and Friability.

ACCEPTANCE CRITERIA

Weight: 0.123 - 0.137 g
Hardness: 2.0 - 8.0 KP
Thickness: 3.0 - 4.0 mm
Friability: NMT 1.0 %
Dissolution: Meets USP Requirement
Disintegration: N/A (for characterization only)
Content Uniformity: 85.0 - 115.0 % TH, (RSD NMT 6.0 %)
Assay: 90.0 - 105.0 % Label

RESULTS - See attached data summary sheets.

AMIDE PHARMACEUTICAL, INC.
PROCESS VALIDATION

DIGOXIN TABLETS, 0.5 mg MPR NO. 14702 - 00

CONCLUSIONS AND COMMENTS

The samples met all acceptance criteria.

The values for weight, hardness, and thickness for the three batches were comparable to each other and showed no unusual shifts or trends. The overall averages for weight and hardness are very close to the midpoints of the preset ranges. Therefore, no revisions to these limits are indicated by the validation data. The midpoint of the thickness range is 3.5 mm, while the observed value for these three batches is 3.3 mm. Since the variability is so low the guideline should be met without difficulty. Therefore the range will not be revised at this time. However, if this trend continues during future production, the range will be revised when sufficient data is obtained. Results are attached in both tabular and graphical form.

Content Uniformity was within limits for all samples tested, with no significant trends being observed. All values except one were within 95 - 107 % L. One tablet had a value of 88, which is within the limit. The values obtained were observed to agree favorably with the blend assays. It should be noted that the averages for the blend assays, and the content uniformity results are essentially the label amount.

All Dissolution samples for the three batches met the USP requirements. This statement is true for both USP XXII (60 Min.) and XXIII (15 & 60 Min.). The values for the three batches were comparable, however there is some variability within the batches themselves. This is more pronounced in the 60 minute readings than in the 15. Batch 4301A appears to be less variable than the other two.

Friability values were all well within the acceptance criteria, and comparable for the three batches.

Disintegration results were comparable with no unusual shifts or trends. Note that this test was run for characterization only, and therefore no acceptance criteria have been, or will be, established.

Acceptable tablets were produced at the low press speed

**AMIDE PHARMACEUTICAL, INC.
PROCESS VALIDATION**

DIGOXIN TABLETS, 0.5 mg MPR NO. 14702 - 00

Acceptable tablets were produced at the low press speed for all three batches, and at high speed for batch 4296A. Unacceptable tablets (weight variability) were produced at high speed for batches 4300A, and 4301A. The normal, high, and low operating speeds for each batch are as follows. Based on the data obtained here, the press may be safely run as slow as 17 rpm. No upper limit can be set at this time.

BATCH	NORMAL	HIGH	LOW
4296A	22 rpm	27 rpm	17 rpm
4300A	22 rpm	27 rpm	17 rpm
4301A	22 rpm	28 rpm	17 rpm

The high and low hardness validation produced acceptable tablets at both ends of the range. Tablets with hardness above the upper limit could not be produced. Therefore the guideline will remain at 2.0 - 8.0 KP. The values for friability are listed below. Those for dissolution are attached.

		FRIABILITY (%)		
	BATCH	4296A	4300A	4301A
LOW KP	FRONT	0.06	0.04	0.05
	REAR	0.05	0.05	0.06
HIGH KP	FRONT	0.03	0.02	0.04
	REAR	0.03	0.03	0.03

The results for the overall composites are attached. These are also all within the acceptance criteria, and are essentially comparable to those obtained for the individual samples.

AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg - Batch # 4296A**

Compression - Weight (g) - Front

Date	Time	1	2	3	4	5	6	7	8	9	10	Average	St. Dev.	RSD
10/17/94	3:00 PM	0.129	0.131	0.130	0.129	0.130	0.133	0.130	0.132	0.129	0.132	0.131	0.001	1.1
10/17/94	3:15 PM	0.131	0.132	0.129	0.130	0.131	0.132	0.132	0.131	0.132	0.130	0.131	0.001	0.8
10/17/94	3:30 PM	0.132	0.131	0.131	0.133	0.131	0.132	0.129	0.131	0.130	0.133	0.131	0.001	1.0
10/17/94	3:45 PM	0.136	0.133	0.135	0.131	0.133	0.132	0.133	0.134	0.131	0.130	0.133	0.002	1.4
10/17/94	4:00 PM	0.130	0.128	0.130	0.133	0.126	0.130	0.131	0.131	0.128	0.131	0.130	0.002	1.5
10/17/94	4:15 PM	0.129	0.129	0.126	0.129	0.127	0.128	0.128	0.129	0.129	0.128	0.128	0.001	0.8
10/17/94	4:30 PM	0.129	0.131	0.131	0.129	0.128	0.128	0.131	0.128	0.129	0.128	0.129	0.001	1.0
10/17/94	4:45 PM	0.130	0.129	0.131	0.131	0.130	0.129	0.130	0.132	0.131	0.131	0.130	0.001	0.7
10/17/94	5:00 PM	0.127	0.134	0.131	0.133	0.131	0.130	0.129	0.129	0.132	0.128	0.130	0.002	1.7
10/17/94	5:15 PM	0.128	0.130	0.129	0.128	0.129	0.131	0.133	0.132	0.130	0.129	0.130	0.002	1.3
10/18/94	8:15 AM	0.128	0.127	0.128	0.133	0.132	0.131	0.130	0.129	0.129	0.132	0.130	0.002	1.6
10/18/94	8:30 AM	0.130	0.130	0.130	0.129	0.130	0.129	0.130	0.129	0.129	0.130	0.130	0.001	0.4

Compression - Weight (g) - Rear

Date	Time	1	2	3	4	5	6	7	8	9	10	Average	St. Dev.	RSD
10/17/94	3:00 PM	0.131	0.130	0.131	0.130	0.130	0.129	0.131	0.132	0.130	0.131	0.131	0.001	0.7
10/17/94	3:15 PM	0.130	0.130	0.131	0.131	0.130	0.130	0.131	0.131	0.129	0.131	0.130	0.001	0.5
10/17/94	3:30 PM	0.132	0.129	0.131	0.127	0.129	0.130	0.129	0.132	0.129	0.130	0.130	0.002	1.2
10/17/94	3:45 PM	0.132	0.137	0.128	0.132	0.130	0.133	0.132	0.132	0.130	0.131	0.132	0.002	1.8
10/17/94	4:00 PM	0.131	0.131	0.127	0.131	0.130	0.129	0.132	0.131	0.130	0.130	0.130	0.001	1.1
10/17/94	4:15 PM	0.131	0.129	0.131	0.130	0.132	0.132	0.130	0.131	0.130	0.131	0.131	0.001	0.7
10/17/94	4:30 PM	0.130	0.130	0.129	0.129	0.131	0.129	0.130	0.132	0.131	0.130	0.130	0.001	0.8
10/17/94	4:45 PM	0.130	0.131	0.131	0.130	0.131	0.131	0.131	0.130	0.129	0.130	0.130	0.001	0.5
10/17/94	5:00 PM	0.133	0.132	0.133	0.131	0.130	0.131	0.132	0.129	0.130	0.132	0.131	0.001	1.0
10/17/94	5:15 PM	0.131	0.132	0.131	0.133	0.132	0.133	0.132	0.131	0.132	0.130	0.132	0.001	0.7
10/18/94	8:15 AM	0.130	0.130	0.129	0.128	0.129	0.131	0.129	0.129	0.128	0.129	0.129	0.001	0.7
10/18/94	8:30 AM	0.129	0.132	0.130	0.131	0.130	0.132	0.130	0.131	0.132	0.131	0.131	0.001	0.8

ANIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIQUEEN TABLETS, 0.5 mg - Batch # 4300A**

Compression - Weight (g) - Front

Date	Time	1	2	3	4	5	6	7	8	9	10	Average	St. Dev.	RSD
10/20/94	3:45 PM	0.132	0.131	0.133	0.133	0.135	0.132	0.134	0.135	0.133	0.134	0.133	0.001	1.0
10/20/94	4:00 PM	0.131	0.134	0.133	0.131	0.132	0.133	0.132	0.133	0.132	0.133	0.132	0.001	0.7
10/20/94	4:15 PM	0.135	0.131	0.134	0.131	0.133	0.134	0.133	0.134	0.134	0.134	0.133	0.001	1.0
10/20/94	4:30 PM	0.133	0.134	0.132	0.133	0.133	0.132	0.132	0.133	0.131	0.132	0.132	0.001	0.6
10/20/94	4:45 PM	0.131	0.133	0.131	0.130	0.132	0.131	0.133	0.129	0.135	0.133	0.132	0.002	1.3
10/20/94	5:00 PM	0.134	0.130	0.130	0.129	0.129	0.130	0.128	0.129	0.130	0.130	0.130	0.002	1.2
10/20/94	5:15 PM	0.129	0.132	0.132	0.130	0.130	0.129	0.130	0.133	0.132	0.127	0.130	0.002	1.2
10/20/94	5:30 PM	0.130	0.130	0.132	0.131	0.130	0.130	0.130	0.132	0.129	0.131	0.131	0.001	0.9
10/20/94	5:45 PM	0.132	0.131	0.130	0.131	0.131	0.131	0.130	0.132	0.129	0.131	0.131	0.001	0.7
10/21/94	7:45 AM	0.132	0.132	0.129	0.131	0.131	0.131	0.130	0.131	0.131	0.131	0.131	0.001	0.7
10/21/94	8:45 AM	0.128	0.129	0.132	0.129	0.133	0.131	0.129	0.130	0.131	0.130	0.130	0.002	1.2
10/21/94	9:00 AM	0.130	0.131	0.130	0.132	0.131	0.133	0.131	0.129	0.132	0.129	0.131	0.001	1.0

Compression - Weight (g) - Rear

Date	Time	1	2	3	4	5	6	7	8	9	10	Average	St. Dev.	RSD
10/20/94	3:45 PM	0.133	0.132	0.130	0.133	0.132	0.131	0.130	0.132	0.131	0.131	0.132	0.001	0.8
10/20/94	4:00 PM	0.131	0.133	0.129	0.132	0.131	0.129	0.131	0.133	0.131	0.132	0.131	0.001	1.1
10/20/94	4:15 PM	0.128	0.130	0.130	0.131	0.131	0.132	0.131	0.130	0.132	0.131	0.131	0.001	0.9
10/20/94	4:30 PM	0.130	0.131	0.131	0.132	0.131	0.128	0.132	0.130	0.130	0.132	0.131	0.001	1.0
10/20/94	4:45 PM	0.132	0.130	0.132	0.131	0.131	0.129	0.130	0.131	0.131	0.132	0.131	0.001	0.8
10/20/94	5:00 PM	0.130	0.133	0.132	0.134	0.133	0.134	0.130	0.131	0.130	0.133	0.132	0.002	1.2
10/20/94	5:15 PM	0.132	0.132	0.131	0.131	0.132	0.131	0.132	0.132	0.131	0.132	0.132	0.001	0.4
10/20/94	5:30 PM	0.130	0.132	0.131	0.131	0.133	0.132	0.131	0.130	0.129	0.128	0.131	0.001	1.1
10/20/94	5:45 PM	0.133	0.131	0.132	0.131	0.132	0.131	0.130	0.132	0.127	0.131	0.131	0.002	1.2
10/21/94	7:45 AM	0.134	0.129	0.130	0.131	0.132	0.131	0.130	0.131	0.132	0.133	0.131	0.001	1.1
10/21/94	8:45 AM	0.130	0.130	0.133	0.133	0.129	0.129	0.129	0.129	0.130	0.131	0.130	0.002	1.2
10/21/94	9:00 AM	0.129	0.129	0.129	0.133	0.132	0.129	0.131	0.129	0.132	0.128	0.130	0.002	1.3

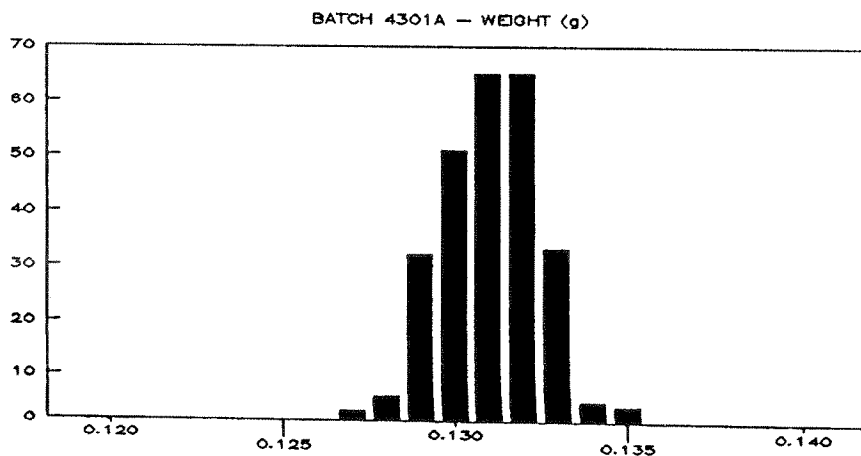
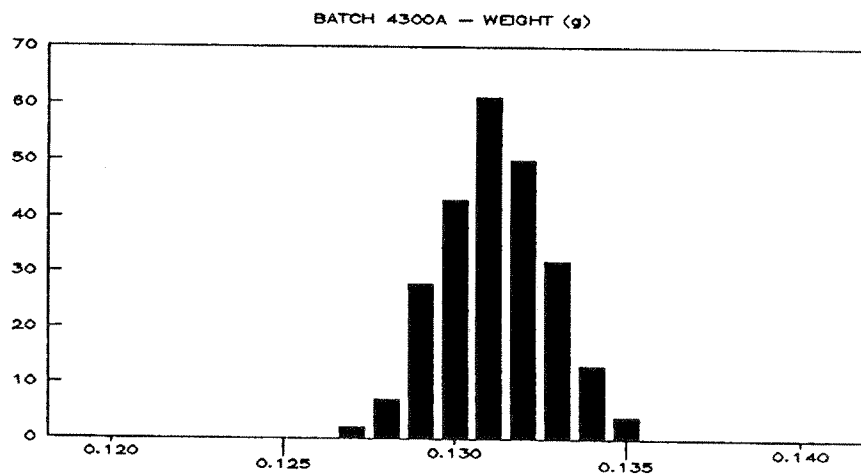
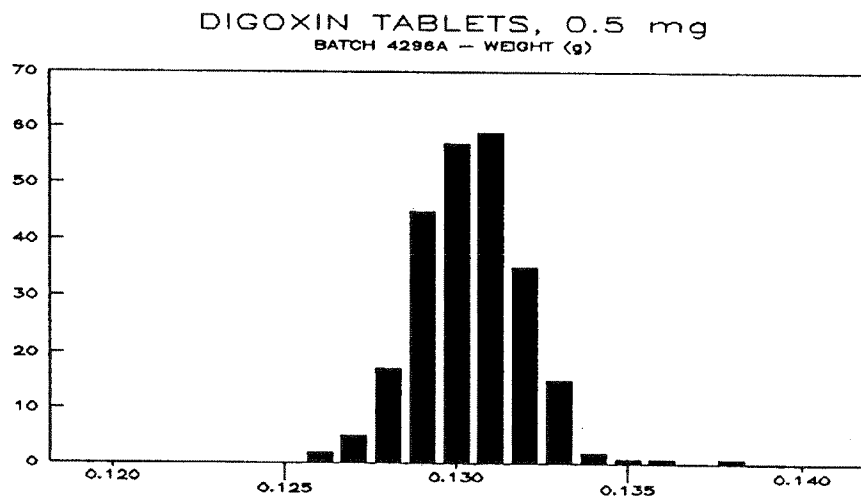
AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg - Batch # 4301A**

Compression - Weight (g) - Front

Date	Time	1	2	3	4	5	6	7	8	9	10	Average	St Dev.	RSD
10/21/94	11:15 AM	0.131	0.132	0.130	0.129	0.132	0.130	0.130	0.131	0.129	0.131	0.131	0.001	0.8
10/21/94	11:30 AM	0.129	0.131	0.132	0.129	0.131	0.132	0.131	0.129	0.128	0.130	0.130	0.001	1.1
10/21/94	11:45 AM	0.133	0.132	0.133	0.132	0.131	0.133	0.132	0.131	0.132	0.132	0.132	0.001	0.6
10/21/94	12:00 PM	0.132	0.133	0.131	0.129	0.133	0.132	0.131	0.133	0.132	0.131	0.132	0.001	1.0
10/21/94	12:15 PM	0.130	0.133	0.130	0.129	0.131	0.131	0.131	0.131	0.131	0.131	0.131	0.001	0.8
10/21/94	12:30 PM	0.131	0.131	0.132	0.130	0.132	0.133	0.130	0.130	0.130	0.132	0.131	0.001	0.8
10/21/94	12:45 PM	0.132	0.130	0.132	0.133	0.131	0.129	0.134	0.132	0.130	0.131	0.131	0.002	1.1
10/21/94	1:00 PM	0.130	0.132	0.128	0.130	0.130	0.129	0.130	0.132	0.131	0.130	0.130	0.001	0.9
10/21/94	1:15 PM	0.129	0.130	0.130	0.129	0.132	0.131	0.130	0.130	0.130	0.129	0.130	0.001	0.7
10/21/94	1:30 PM	0.130	0.130	0.131	0.130	0.133	0.131	0.132	0.130	0.129	0.133	0.131	0.001	1.0
10/21/94	3:05 PM	0.131	0.131	0.128	0.132	0.130	0.132	0.132	0.132	0.131	0.135	0.131	0.002	1.4
10/21/94	3:20 PM	0.132	0.131	0.129	0.131	0.129	0.131	0.132	0.130	0.129	0.131	0.131	0.001	0.9
10/21/94	3:35 PM	0.131	0.130	0.131	0.127	0.132	0.130	0.131	0.129	0.128	0.129	0.130	0.002	1.2

Compression - Weight (g) - Rear

Date	Time	1	2	3	4	5	6	7	8	9	10	Average	St Dev.	RSD
10/21/94	11:15 AM	0.129	0.130	0.129	0.130	0.130	0.131	0.132	0.129	0.130	0.129	0.130	0.001	0.8
10/21/94	11:30 AM	0.130	0.133	0.132	0.131	0.132	0.130	0.130	0.129	0.131	0.132	0.131	0.001	1.0
10/21/94	11:45 AM	0.131	0.133	0.131	0.133	0.132	0.133	0.132	0.131	0.131	0.130	0.132	0.001	0.8
10/21/94	12:00 PM	0.133	0.133	0.132	0.131	0.133	0.129	0.131	0.133	0.132	0.130	0.132	0.001	1.1
10/21/94	12:15 PM	0.135	0.132	0.132	0.132	0.131	0.131	0.133	0.132	0.133	0.132	0.132	0.001	0.9
10/21/94	12:30 PM	0.131	0.132	0.133	0.134	0.132	0.132	0.133	0.134	0.132	0.132	0.133	0.001	0.7
10/21/94	12:45 PM	0.133	0.132	0.132	0.133	0.132	0.130	0.133	0.132	0.131	0.133	0.132	0.001	0.8
10/21/94	1:00 PM	0.132	0.131	0.135	0.131	0.130	0.130	0.131	0.131	0.132	0.128	0.131	0.002	1.4
10/21/94	1:15 PM	0.132	0.133	0.132	0.129	0.133	0.132	0.130	0.132	0.131	0.133	0.132	0.001	1.0
10/21/94	1:30 PM	0.129	0.133	0.134	0.130	0.132	0.129	0.132	0.129	0.130	0.129	0.131	0.002	1.4
10/21/94	3:05 PM	0.131	0.132	0.129	0.130	0.132	0.131	0.131	0.133	0.132	0.132	0.131	0.001	0.9
10/21/94	3:20 PM	0.131	0.132	0.131	0.131	0.130	0.131	0.131	0.130	0.129	0.131	0.131	0.001	0.6
10/21/94	3:35 PM	0.133	0.131	0.131	0.130	0.130	0.129	0.131	0.127	0.131	0.132	0.131	0.002	1.3



AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg - Batch # 4296A**

Compression - Hardness (kp) - Front

Date	Time	1	2	3	4	5	Average	St Dev.	RSD
10/17/94	3:00 PM	4.6	3.8	4.6	3.9	4.6	4.3	0.4	9.6
10/17/94	3:15 PM	4.7	4.2	4.5	4.2	4.7	4.5	0.3	5.6
10/17/94	3:30 PM	4.6	4.7	5.0	4.7	5.0	4.8	0.2	3.9
10/17/94	3:45 PM	5.0	4.4	4.5	4.1	5.0	4.6	0.4	8.6
10/17/94	4:00 PM	3.8	4.2	3.9	4.3	4.2	4.1	0.2	5.3
10/17/94	4:15 PM	3.9	3.9	4.0	3.7	3.9	3.9	0.1	2.8
10/17/94	4:30 PM	4.5	3.8	4.1	4.6	4.1	4.2	0.3	7.8
10/17/94	4:45 PM	4.1	3.9	4.2	4.5	4.7	4.3	0.3	7.5
10/17/94	5:00 PM	4.5	3.9	4.0	4.1	4.8	4.3	0.4	8.9
10/17/94	5:15 PM	4.9	4.3	3.9	4.0	4.1	4.2	0.4	9.4
10/18/94	8:15 AM	4.0	3.6	4.3	4.0	4.0	4.0	0.2	6.3
10/18/94	8:30 PM	4.3	4.0	4.0	4.2	4.1	4.1	0.1	3.2

Compression - Hardness (kp) - Rear

Date	Time	1	2	3	4	5	Average	St Dev.	RSD
10/17/94	3:00 PM	4.3	4.0	3.9	4.1	6.9	4.6	1.3	27.4
10/17/94	3:15 PM	4.2	4.3	3.7	4.2	4.6	4.2	0.3	7.7
10/17/94	3:30 PM	4.3	4.1	4.3	3.8	4.5	4.2	0.3	6.3
10/17/94	3:45 PM	4.0	4.6	4.7	4.0	3.9	4.2	0.4	8.9
10/17/94	4:00 PM	3.8	3.8	4.1	4.3	4.2	4.0	0.2	5.7
10/17/94	4:15 PM	4.5	4.6	4.1	4.4	4.0	4.3	0.3	6.0
10/17/94	4:30 PM	4.3	4.3	4.4	4.2	4.3	4.3	0.1	1.6
10/17/94	4:45 PM	4.6	4.2	4.0	4.1	4.1	4.2	0.2	5.6
10/17/94	5:00 PM	4.5	4.4	4.5	4.1	4.7	4.4	0.2	4.9
10/17/94	5:15 PM	4.6	4.8	4.4	4.6	4.5	4.6	0.1	3.2
10/18/94	8:15 AM	3.7	3.6	3.6	3.9	3.5	3.7	0.2	4.1
10/18/94	8:30 PM	3.7	4.1	3.8	4.2	4.1	4.0	0.2	5.4

AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg - Batch # 4300A**

Compression - Hardness (kp) - Front

Date	Time	1	2	3	4	5	Average	St Dev.	RSD
10/20/94	3:45 PM	4.8	5.0	4.9	4.7	5.0	4.9	0.1	2.7
10/20/94	4:00 PM	5.3	4.4	4.6	4.4	4.9	4.7	0.4	8.1
10/20/94	4:15 PM	5.3	5.1	5.2	5.0	5.2	5.2	0.1	2.2
10/20/94	4:30 PM	5.0	5.0	4.9	5.2	5.1	5.0	0.1	2.3
10/20/94	4:45 PM	5.0	5.3	4.5	4.8	5.1	4.9	0.3	6.2
10/20/94	5:00 PM	4.4	4.3	4.4	4.8	4.7	4.5	0.2	4.8
10/20/94	5:15 PM	4.9	4.4	5.3	4.6	4.6	4.8	0.4	7.4
10/20/94	5:30 PM	5.2	4.9	5.1	5.2	5.0	5.1	0.1	2.6
10/20/94	5:45 PM	5.2	5.3	5.1	4.7	4.8	5.0	0.3	5.2
10/21/94	7:45 AM	4.6	5.3	4.7	4.7	4.6	4.8	0.3	6.2
10/21/94	8:45 AM	4.6	4.7	4.4	4.7	4.6	4.6	0.1	2.7
10/21/94	9:00 AM	5.1	5.1	4.9	4.8	4.9	5.0	0.1	2.7

Compression - Hardness (kp) - Rear

Date	Time	1	2	3	4	5	Average	St Dev.	RSD
10/20/94	3:45 PM	5.2	4.8	4.3	5.2	5.0	4.9	0.4	7.6
10/20/94	4:00 PM	4.8	4.5	5.5	5.0	4.8	4.9	0.4	7.5
10/20/94	4:15 PM	5.8	4.4	4.3	5.0	4.8	4.9	0.6	12.3
10/20/94	4:30 PM	5.0	4.2	4.5	5.4	4.8	4.8	0.5	9.6
10/20/94	4:45 PM	5.0	4.8	5.1	5.0	5.0	5.0	0.1	2.2
10/20/94	5:00 PM	5.1	4.7	5.2	4.7	5.0	4.9	0.2	4.7
10/20/94	5:15 PM	5.3	5.2	5.7	5.1	5.1	5.3	0.2	4.7
10/20/94	5:30 PM	4.9	4.7	5.7	5.1	4.8	5.0	0.4	7.9
10/20/94	5:45 PM	5.0	5.2	5.3	4.0	4.7	4.8	0.5	10.8
10/21/94	7:45 AM	5.1	4.8	4.6	4.7	4.9	4.8	0.2	4.0
10/21/94	8:45 AM	5.2	4.5	3.9	4.9	4.7	4.6	0.5	10.5
10/21/94	9:00 AM	4.6	5.4	5.0	4.8	4.8	4.9	0.3	6.2

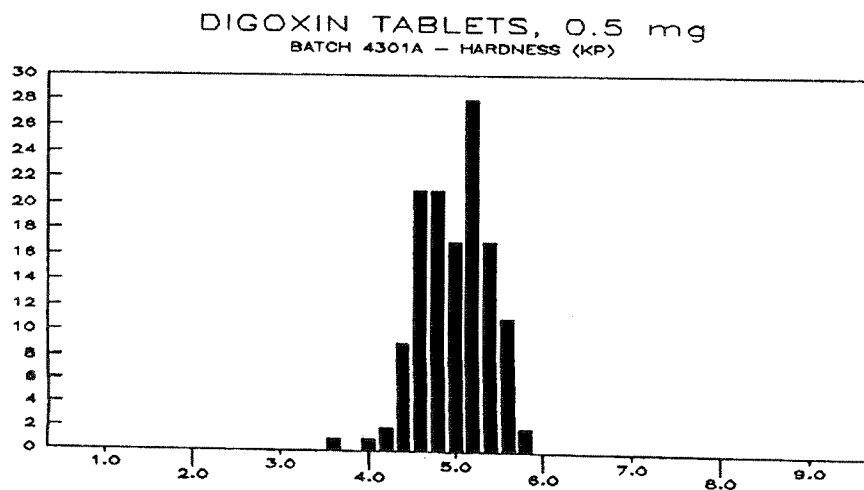
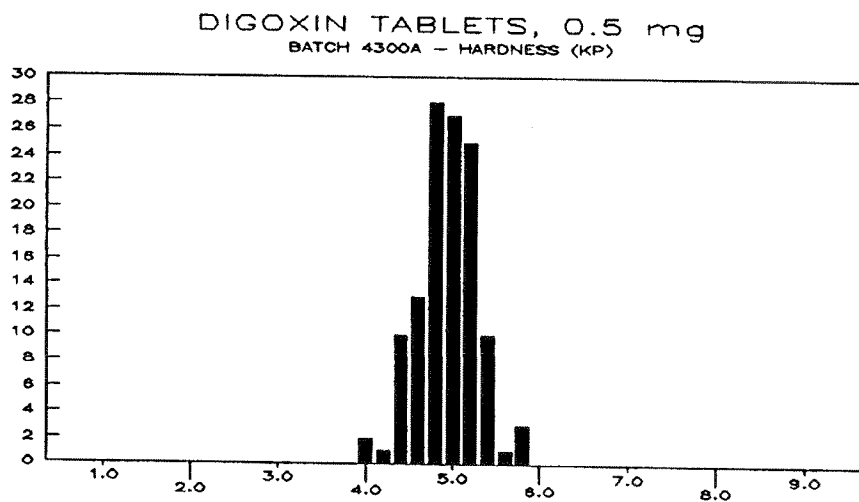
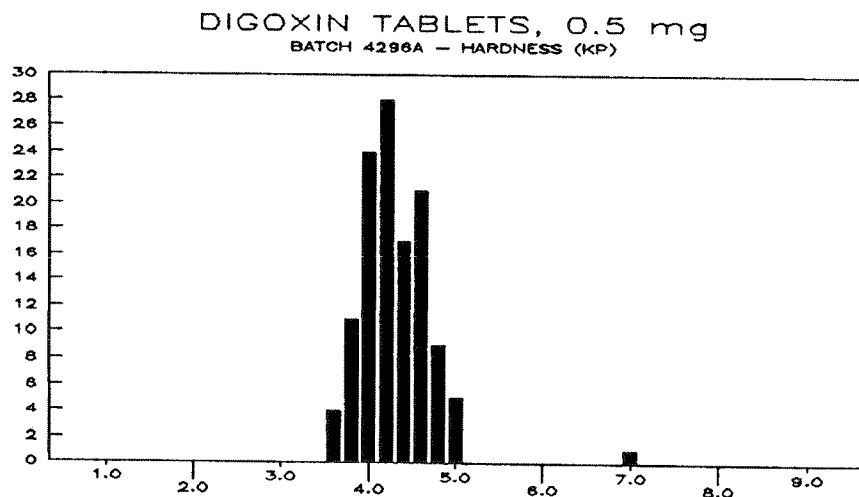
AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg - Batch # 4301A**

Compression - Hardness (kp) - Front

Date	Time	1	2	3	4	5	Average	St Dev.	RSD
10/21/94	11:15 AM	4.7	5.0	5.0	4.7	4.8	4.8	0.2	3.1
10/21/94	11:30 AM	4.6	4.8	5.0	5.2	4.9	4.9	0.2	4.6
10/21/94	11:45 AM	5.3	5.1	5.2	5.5	5.3	5.3	0.1	2.8
10/21/94	12:00 PM	5.5	5.4	4.8	5.3	5.4	5.3	0.3	5.3
10/21/94	12:15 PM	4.7	5.1	4.6	4.6	5.1	4.8	0.3	5.4
10/21/94	12:30 PM	4.5	5.2	4.7	4.8	5.1	4.9	0.3	5.9
10/21/94	12:45 PM	5.0	5.1	4.9	5.2	5.2	5.1	0.1	2.6
10/21/94	1:00 PM	4.6	4.4	5.0	4.5	4.4	4.6	0.2	5.4
10/21/94	1:15 PM	4.8	4.9	5.1	4.9	4.6	4.9	0.2	3.7
10/21/94	1:30 PM	4.7	4.6	5.1	4.9	5.1	4.9	0.2	4.7
10/21/94	3:05 PM	4.0	3.6	4.1	4.4	4.6	4.1	0.4	9.3
10/21/94	3:20 PM	4.7	4.5	4.5	4.2	4.6	4.5	0.2	4.2
10/21/94	3:35 PM	4.3	4.5	4.8	4.7	4.3	4.5	0.2	5.0

Compression - Hardness (kp) - Rear

Date	Time	1	2	3	4	5	Average	St Dev.	RSD
10/21/94	11:15 AM	5.0	5.1	4.9	5.1	4.4	4.9	0.3	5.9
10/21/94	11:30 AM	5.2	5.5	5.3	5.1	4.8	5.2	0.3	5.0
10/21/94	11:45 AM	5.5	5.6	4.8	5.4	5.5	5.4	0.3	6.0
10/21/94	12:00 PM	5.5	5.1	5.6	5.7	4.9	5.4	0.3	6.4
10/21/94	12:15 PM	5.1	5.3	5.5	5.2	5.3	5.3	0.1	2.8
10/21/94	12:30 PM	5.0	4.8	5.7	5.2	5.3	5.2	0.3	6.5
10/21/94	12:45 PM	5.4	5.4	5.3	5.5	5.3	5.4	0.1	1.6
10/21/94	1:00 PM	4.4	5.1	4.6	5.3	5.1	4.9	0.4	7.8
10/21/94	1:15 PM	5.2	5.4	5.2	5.2	5.3	5.3	0.1	1.7
10/21/94	1:30 PM	4.5	4.9	5.0	4.6	5.5	4.9	0.4	8.0
10/21/94	3:05 PM	4.5	4.7	4.5	4.4	4.4	4.5	0.1	2.7
10/21/94	3:20 PM	4.6	4.8	4.8	4.6	4.8	4.7	0.1	2.3
10/21/94	3:35 PM	4.5	5.0	5.1	5.1	4.8	4.9	0.3	5.2



AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg - Batch # 4296A**

Compression - Thickness (mm) - Front

Date	Time	1	2	3	4	5	Average	St Dev.	RSD
10/17/94	3:00 PM	3.36	3.38	3.40	3.35	3.35	3.37	0.02	0.6
10/17/94	3:15 PM	3.39	3.37	3.36	3.38	3.37	3.37	0.01	0.3
10/17/94	3:30 PM	3.38	3.36	3.38	3.37	3.38	3.37	0.01	0.3
10/17/94	3:45 PM	3.36	3.40	3.42	3.40	3.38	3.39	0.02	0.7
10/17/94	4:00 PM	3.33	3.34	3.34	3.35	3.34	3.34	0.01	0.2
10/17/94	4:15 PM	3.33	3.33	3.33	3.34	3.34	3.33	0.01	0.2
10/17/94	4:30 PM	3.32	3.33	3.37	3.34	3.35	3.34	0.02	0.6
10/17/94	4:45 PM	3.35	3.34	3.34	3.35	3.35	3.35	0.01	0.2
10/17/94	5:00 PM	3.33	3.33	3.35	3.36	3.35	3.34	0.01	0.4
10/17/94	5:15 PM	3.35	3.32	3.34	3.32	3.36	3.34	0.02	0.5
10/18/94	8:15 AM	3.33	3.35	3.38	3.35	3.39	3.36	0.02	0.7
10/18/94	8:30 AM	3.35	3.33	3.37	3.37	3.34	3.35	0.02	0.5

Compression - Thickness (mm) - Rear

Date	Time	1	2	3	4	5	Average	St Dev.	RSD
10/17/94	3:00 PM	3.28	3.40	3.36	3.38	3.37	3.36	0.05	1.4
10/17/94	3:15 PM	3.35	3.37	3.36	3.38	3.36	3.36	0.01	0.3
10/17/94	3:30 PM	3.36	3.38	3.40	3.38	3.35	3.37	0.02	0.6
10/17/94	3:45 PM	3.40	3.38	3.36	3.37	3.36	3.37	0.02	0.5
10/17/94	4:00 PM	3.35	3.35	3.36	3.34	3.35	3.35	0.01	0.2
10/17/94	4:15 PM	3.38	3.36	3.36	3.38	3.34	3.36	0.02	0.5
10/17/94	4:30 PM	3.33	3.34	3.34	3.34	3.33	3.34	0.01	0.2
10/17/94	4:45 PM	3.34	3.35	3.36	3.35	3.32	3.34	0.02	0.5
10/17/94	5:00 PM	3.34	3.37	3.36	3.40	3.35	3.36	0.02	0.7
10/17/94	5:15 PM	3.37	3.36	3.38	3.35	3.39	3.37	0.02	0.5
10/18/94	8:15 AM	3.35	3.33	3.37	3.36	3.36	3.35	0.02	0.5
10/18/94	8:30 AM	3.36	3.38	3.39	3.37	3.36	3.37	0.01	0.4

AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg - Batch # 4300A**

Compression - Thickness (mm) - Front

Date	Time	1	2	3	4	5	Average	St. Dev.	RSD
10/20/94	3:45 PM	3.37	3.38	3.37	3.39	3.38	3.38	0.01	0.2
10/20/94	4:00 PM	3.37	3.38	3.40	3.38	3.36	3.38	0.01	0.4
10/20/94	4:15 PM	3.36	3.39	3.41	3.36	3.35	3.37	0.03	0.7
10/20/94	4:30 PM	3.35	3.33	3.37	3.39	3.36	3.36	0.02	0.7
10/20/94	4:45 PM	3.36	3.35	3.34	3.38	3.32	3.35	0.02	0.7
10/20/94	5:00 PM	3.30	3.32	3.33	3.31	3.33	3.32	0.01	0.4
10/20/94	5:15 PM	3.30	3.32	3.29	3.32	3.34	3.31	0.02	0.6
10/20/94	5:30 PM	3.33	3.30	3.31	3.32	3.34	3.32	0.02	0.5
10/20/94	5:45 PM	3.28	3.30	3.31	3.34	3.32	3.31	0.02	0.7
10/21/94	7:45 AM	3.30	3.32	3.33	3.31	3.29	3.31	0.02	0.5
10/21/94	8:45 AM	3.36	3.35	3.33	3.33	3.33	3.34	0.01	0.4
10/21/94	9:00 AM	3.32	3.32	3.35	3.34	3.34	3.33	0.01	0.4

Compression - Thickness (mm) - Rear

Date	Time	1	2	3	4	5	Average	St. Dev.	RSD
10/20/94	3:45 PM	3.36	3.32	3.35	3.33	3.35	3.34	0.02	0.5
10/20/94	4:00 PM	3.33	3.35	3.32	3.30	3.31	3.32	0.02	0.6
10/20/94	4:15 PM	3.34	3.32	3.31	3.34	3.33	3.33	0.01	0.4
10/20/94	4:30 PM	3.30	3.33	3.31	3.32	3.31	3.31	0.01	0.3
10/20/94	4:45 PM	3.31	3.30	3.33	3.29	3.30	3.31	0.02	0.5
10/20/94	5:00 PM	3.32	3.35	3.33	3.35	3.30	3.33	0.02	0.6
10/20/94	5:15 PM	3.34	3.33	3.30	3.33	3.30	3.32	0.02	0.6
10/20/94	5:30 PM	3.30	3.34	3.29	3.28	3.33	3.31	0.03	0.8
10/20/94	5:45 PM	3.33	3.30	3.34	3.31	3.32	3.32	0.02	0.5
10/21/94	7:45 AM	3.33	3.35	3.30	3.38	3.31	3.33	0.03	1.0
10/21/94	8:45 AM	3.30	3.29	3.33	3.31	3.34	3.31	0.02	0.6
10/21/94	9:00 AM	3.37	3.30	3.30	3.32	3.29	3.32	0.03	1.0

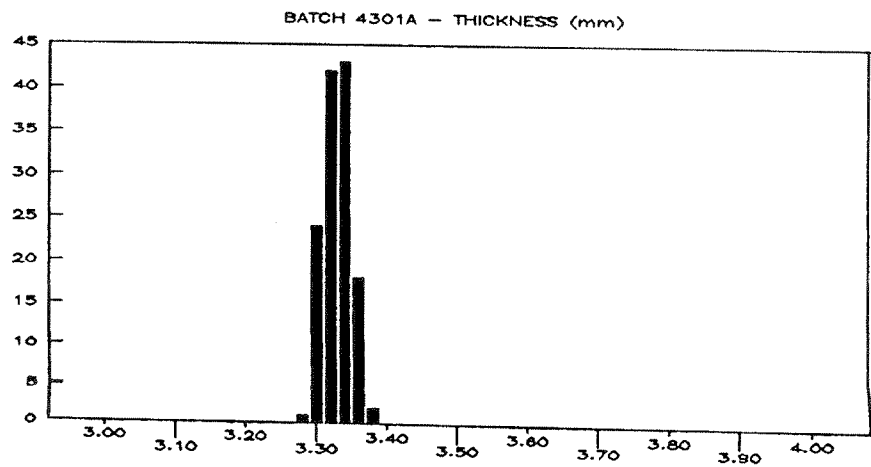
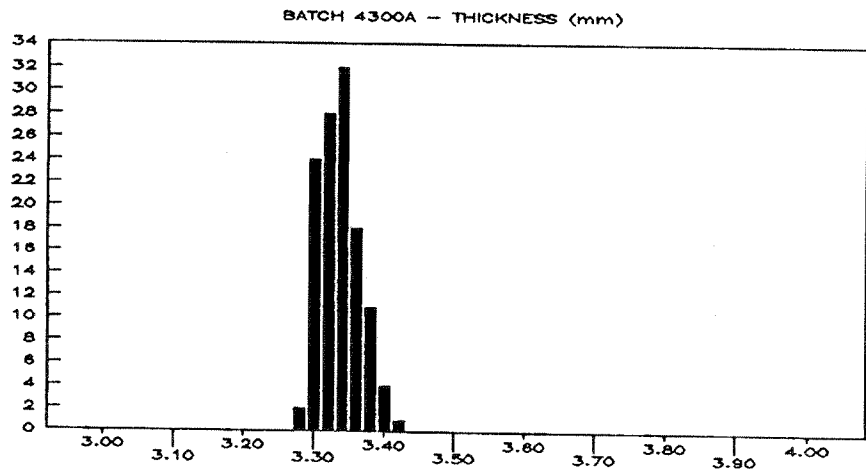
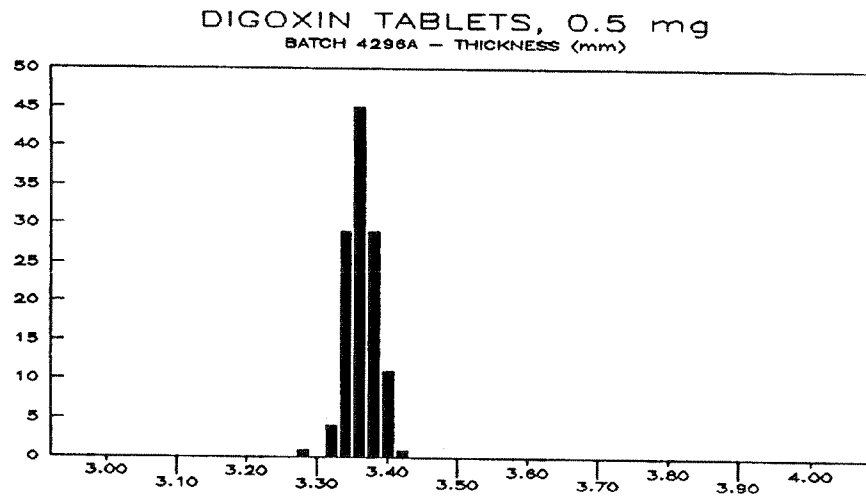
AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg - Batch # 4301A**

Compression - Thickness (mm) - Front

Date	Time	1	2	3	4	5	Average	St Dev.	RSD
10/21/94	11:15 AM	3.30	3.32	3.33	3.30	3.34	3.32	0.02	0.5
10/21/94	11:30 AM	3.33	3.32	3.34	3.30	3.31	3.32	0.02	0.5
10/21/94	11:45 AM	3.31	3.34	3.32	3.33	3.33	3.33	0.01	0.3
10/21/94	12:00 PM	3.31	3.34	3.32	3.33	3.33	3.33	0.01	0.3
10/21/94	12:15 PM	3.32	3.30	3.30	3.30	3.31	3.31	0.01	0.3
10/21/94	12:30 PM	3.36	3.33	3.33	3.32	3.34	3.34	0.02	0.5
10/21/94	12:45 PM	3.30	3.32	3.34	3.31	3.31	3.32	0.02	0.5
10/21/94	1:00 PM	3.31	3.33	3.35	3.32	3.33	3.33	0.01	0.4
10/21/94	1:15 PM	3.30	3.31	3.30	3.30	3.31	3.30	0.01	0.2
10/21/94	1:30 PM	3.31	3.34	3.34	3.35	3.35	3.34	0.02	0.5
10/21/94	3:05 PM	3.33	3.37	3.34	3.33	3.36	3.35	0.02	0.5
10/21/94	3:20 PM	3.35	3.36	3.33	3.34	3.35	3.35	0.01	0.3
10/21/94	3:35 PM	3.32	3.34	3.35	3.30	3.29	3.32	0.03	0.8

Compression - Thickness (mm) - Rear

Date	Time	1	2	3	4	5	Average	St Dev.	RSD
10/21/94	11:15 AM	3.31	3.32	3.35	3.29	3.31	3.32	0.02	0.7
10/21/94	11:30 AM	3.33	3.31	3.30	3.32	3.34	3.32	0.02	0.5
10/21/94	11:45 AM	3.34	3.30	3.32	3.33	3.31	3.32	0.02	0.5
10/21/94	12:00 PM	3.32	3.34	3.31	3.30	3.34	3.32	0.02	0.5
10/21/94	12:15 PM	3.34	3.30	3.34	3.32	3.32	3.32	0.02	0.5
10/21/94	12:30 PM	3.33	3.35	3.34	3.36	3.33	3.34	0.01	0.4
10/21/94	12:45 PM	3.30	3.30	3.31	3.32	3.31	3.31	0.01	0.3
10/21/94	1:00 PM	3.31	3.35	3.33	3.35	3.34	3.34	0.02	0.5
10/21/94	1:15 PM	3.31	3.31	3.32	3.30	3.32	3.31	0.01	0.3
10/21/94	1:30 PM	3.30	3.35	3.30	3.37	3.31	3.33	0.03	1.0
10/21/94	3:05 PM	3.33	3.32	3.36	3.36	3.35	3.34	0.02	0.5
10/21/94	3:20 PM	3.33	3.33	3.34	3.33	3.34	3.33	0.01	0.2
10/21/94	3:35 PM	3.32	3.30	3.30	3.31	3.28	3.30	0.01	0.4



AMIDE PHARMACEUTICAL, INC.

PROCESS VALIDATION

DIGOXIN TABLETS, 0.5 mg

Compression - Content Uniformity (%)

Batch #	4296A	4296A	4300A	4300A	4301A	4301A
Side	Front	Rear	Front	Rear	Front	Rear
1	105.2	104.8	100.1	102.6	97.1	99.2
2	103.3	101.6	99.7	101.6	97.0	100.9
3	103.7	104.5	100.7	102.2	99.6	100.2
4	102.5	102.6	98.5	100.3	100.8	99.8
5	103.6	104.4	100.1	102.0	102.1	99.3
6	104.5	104.0	102.6	100.0	97.9	99.1
7	104.2	102.5	102.2	100.1	101.4	98.0
8	105.9	100.2	102.7	100.0	100.9	102.5
9	97.6	99.9	100.9	98.7	100.5	98.1
10	97.6	98.3	102.6	99.8	101.1	100.2
11	101.1	106.5	103.0	99.3	100.8	100.0
12	101.9	88.0	102.6	100.7	96.1	101.4
13	103.1	99.5	103.3	103.2	101.0	98.2
14	104.3	105.1	101.1	103.4	101.1	100.0
15	101.5	98.9	103.0	102.5	97.1	99.3
16	103.5	99.9	100.9	101.3	100.3	99.4
17	101.0	104.7	100.2	102.0	102.0	98.4
18	100.3	100.4	100.8	99.5	95.3	102.7
19	102.4	99.1	99.5	100.8	99.6	99.4
20	103.1	105.0	99.9	100.2	98.5	99.0
21	101.5	102.1	101.9	99.0	98.4	101.5
22	101.6	99.4	102.3	100.7	99.0	99.8
23	101.5	101.3	103.8	103.0	102.9	100.4
24	104.2	103.7	100.9	99.8	98.1	101.1
25	100.4	103.9	102.3	99.7	99.2	99.4
26	100.9	103.1	100.5	99.8	99.3	99.2
27	101.3	103.0	100.1	99.3	100.9	100.0
28	101.1	102.4	99.5	100.8	99.3	100.6
29	99.4	98.0	99.1	100.0	103.6	99.7
30	98.9	100.0	99.9	99.2	99.1	100.1
Average	102.0	101.6	101.2	100.7	99.7	99.9
St Dev.	2.1	3.5	1.4	1.3	2.0	1.2
RSD	2.0	3.4	1.4	1.3	2.0	1.2

WIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg**

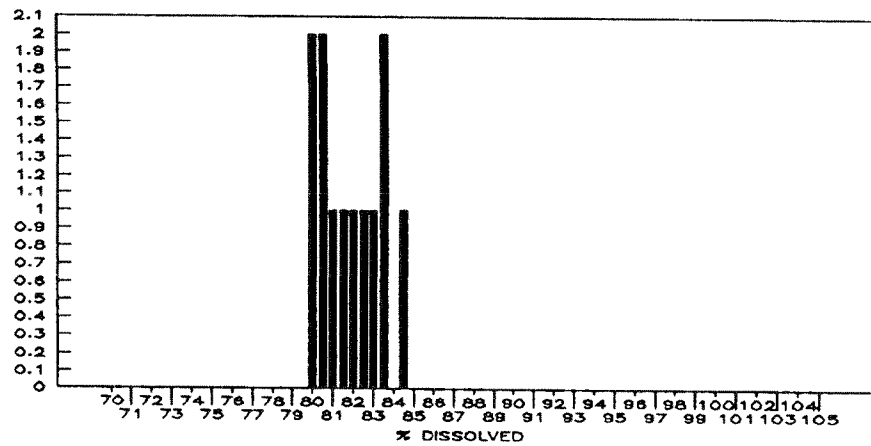
Compression - Dissolution (%) - 15 min.

Batch #	4296A			4300A			4301A		
Sample	1st Third	2nd Third	Final Third	1st Third	2nd Third	Final Third	1st Third	2nd Third	Final Third
1	83.5	79.3	84.1	79.4	75.4	84.4	77.9	77.9	81.2
2	84.2	81.6	80.2	78.0	75.3	79.9	80.6	82.3	80.6
3	81.3	83.4	79.1	82.7	75.4	84.6	78.8	79.0	81.0
4	82.0	82.7	82.4	77.8	75.4	80.9	80.5	77.9	80.5
5	79.8	81.0	82.9	78.1	75.2	80.6	80.5	79.3	81.4
6	80.6	78.7	86.2	75.5	73.1	82.3	79.4	83.7	79.3
7	82.2	82.1	83.3	76.1	82.4	81.8	81.6	81.0	77.6
8	80.1	81.0	83.7	78.8	77.7	86.8	78.6	81.4	78.8
9	83.4	83.1	85.0	76.5	80.9	78.1	83.1	82.2	80.9
10	83.0	82.5	83.9	78.6	79.1	82.2	78.7	78.2	78.5
11	79.7	84.8	87.4	77.4	81.2	79.1	80.3	77.0	79.4
12	80.1	78.2	87.4	75.7	79.5	78.8	79.2	76.9	80.0
Average	81.7	81.5	83.8	77.9	77.6	81.6	79.9	79.7	79.9
St Dev.	1.6	2.0	2.5	2.0	3.0	2.6	1.5	2.3	1.2
RSD	2.0	2.5	3.0	2.5	3.9	3.2	1.8	2.9	1.5

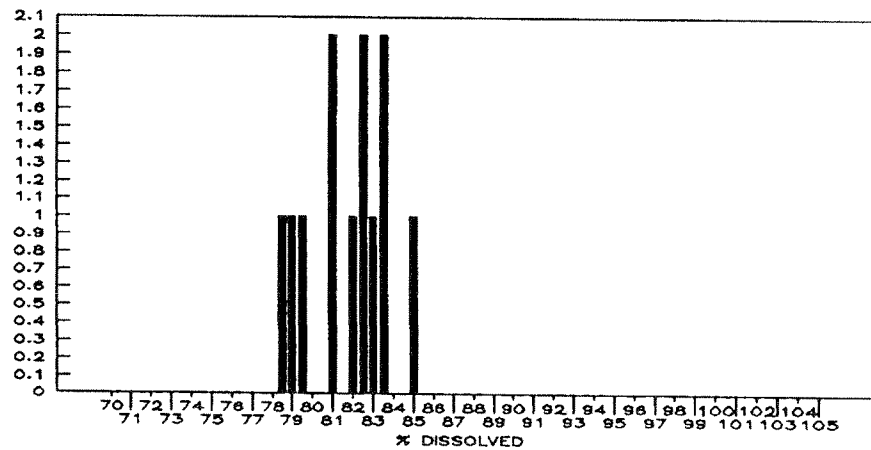
Compression - Dissolution (%) - 60 min.

Batch #	4296A			4300A			4301A		
Sample	1st Third	2nd Third	Final Third	1st Third	2nd Third	Final Third	1st Third	2nd Third	Final Third
1	93.4	87.2	99.7	88.3	91.8	97.7	87.2	99.5	96.9
2	92.9	87.3	101.3	91.3	94.4	89.7	88.4	96.7	98.7
3	91.3	88.8	102.1	91.4	92.4	86.5	90.2	98.8	90.6
4	93.1	87.2	104.4	90.2	92.4	87.6	91.8	92.4	89.4
5	93.6	86.7	103.8	95.4	93.7	87.7	93.4	92.0	95.5
6	92.9	89.9	103.5	89.3	96.4	88.1	97.1	99.3	97.7
7	92.3	87.5	98.4	91.6	95.7	88.4	98.8	88.3	94.4
8	93.4	90.4	94.0	91.4	93.4	88.3	98.3	87.2	91.7
9	91.3	86.1	99.0	90.3	95.1	87.1	101.7	91.9	91.3
10	91.1	83.6	102.7	89.1	92.6	85.6	98.5	98.6	90.3
11	90.5	82.4	105.0	93.4	93.7	83.8	98.8	88.8	91.2
12	100.9	92.8	101.8	94.8	94.3	85.8	99.7	91.2	91.1
Average	93.1	87.5	101.3	91.4	93.8	88.0	95.3	93.7	93.2
St Dev.	2.7	2.8	3.1	2.2	1.4	3.4	4.9	4.6	3.2
RSD	2.9	3.2	3.1	2.4	1.5	3.9	5.1	4.9	3.5

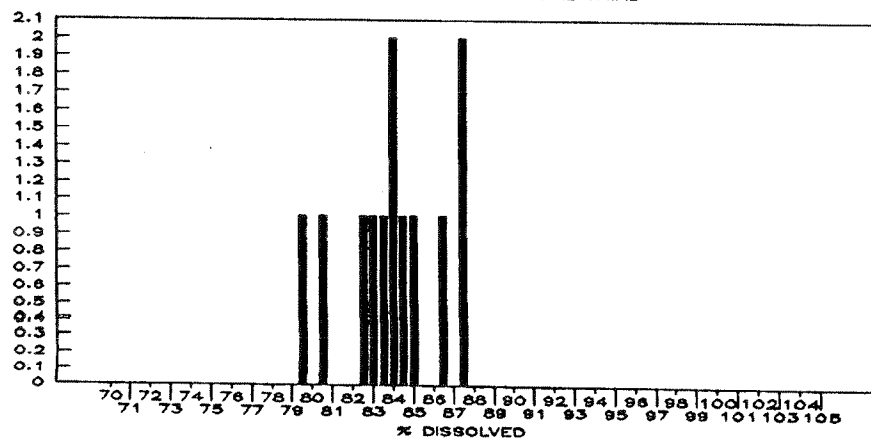
DIGOXIN TABLETS, 0.5 mg — BATCH 4296A
15 MIN. DISSOLUTION — 1st THIRD



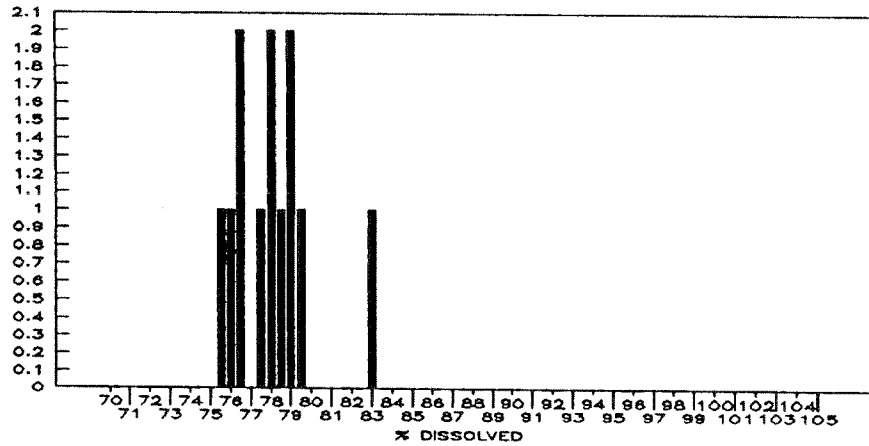
DIGOXIN TABLETS, 0.5 mg — BATCH 4296A
15 MIN. DISSOLUTION — 2nd THIRD



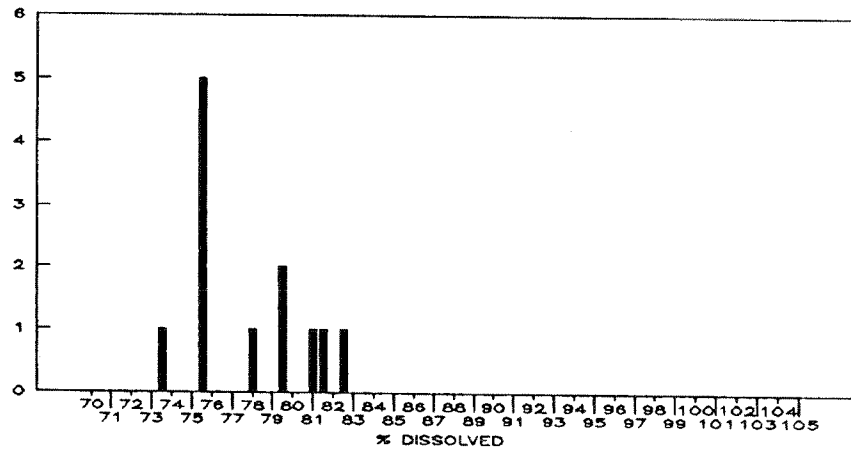
DIGOXIN TABLETS, 0.5 mg — BATCH 4296A
15 MIN. DISSOLUTION — FINAL THIRD



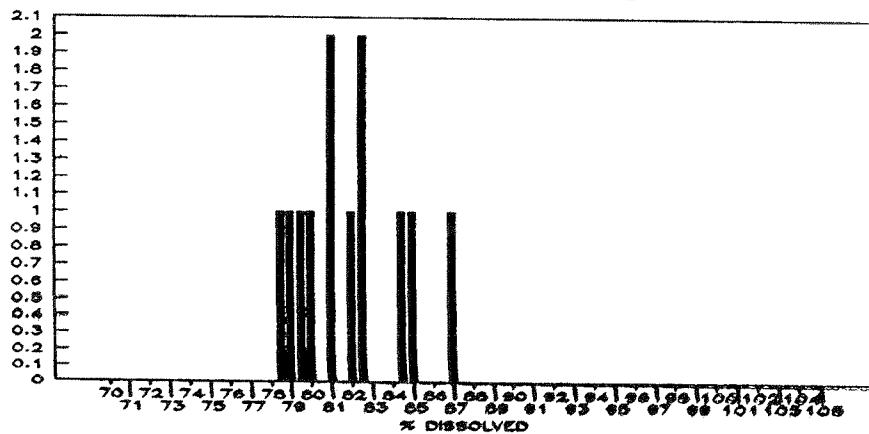
DIGOXIN TABLETS, 0.5 mg — BATCH 4300A
15 MIN. DISSOLUTION — 1st THIRD



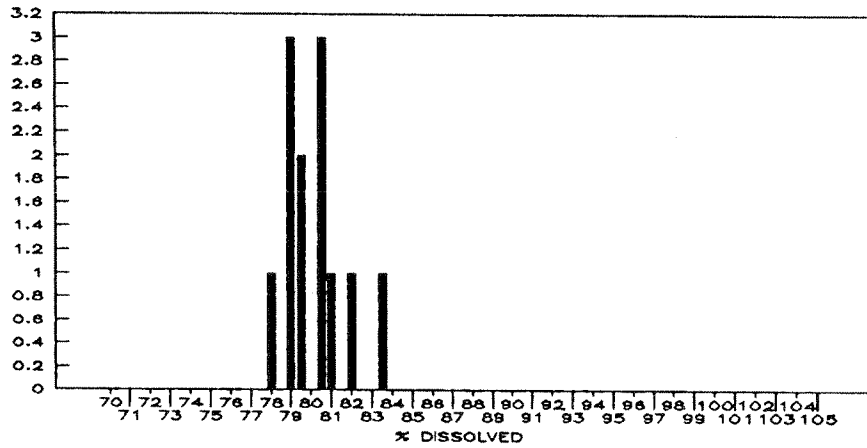
DIGOXIN TABLETS, 0.5 mg — BATCH 4300A
15 MIN. DISSOLUTION — 2nd THIRD



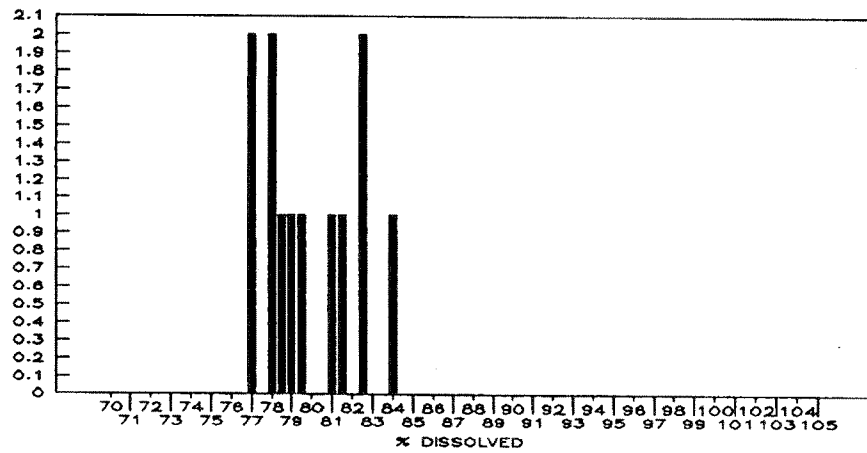
DIGOXIN TABLETS, 0.5 mg — BATCH 4300A
15 MIN. DISSOLUTION — FINAL THIRD



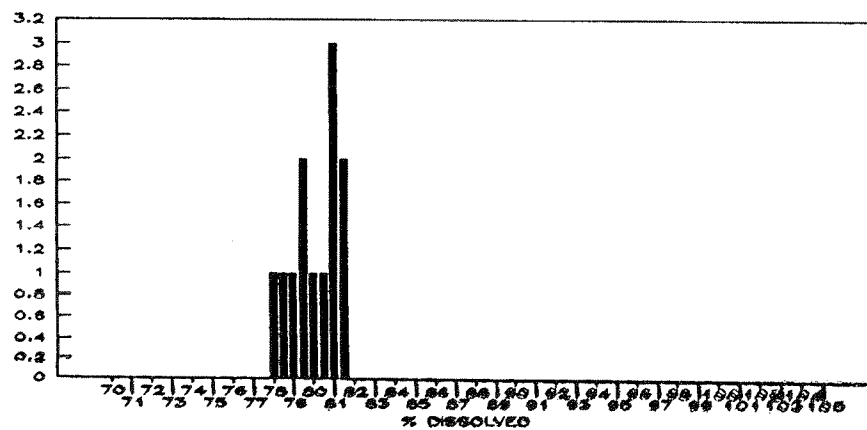
DIGOXIN TABLETS, 0.5 mg — BATCH 4301A
15 MIN. DISSOLUTION — 1st THIRD



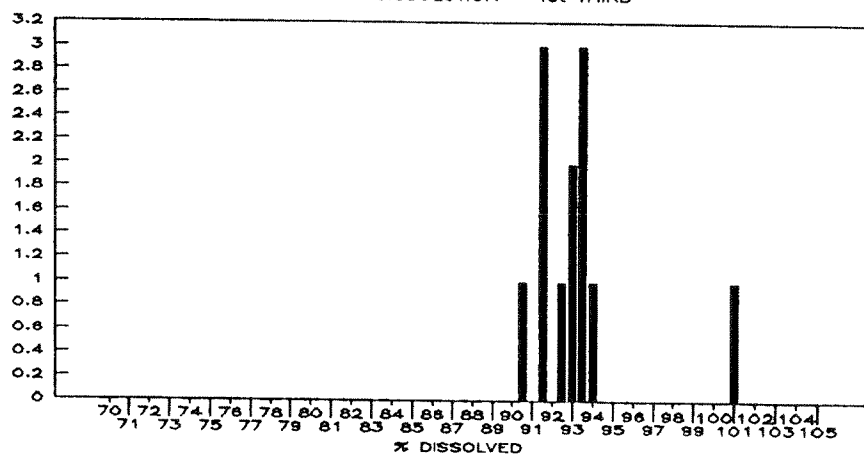
DIGOXIN TABLETS, 0.5 mg — BATCH 4301A
15 MIN. DISSOLUTION — 2nd THIRD



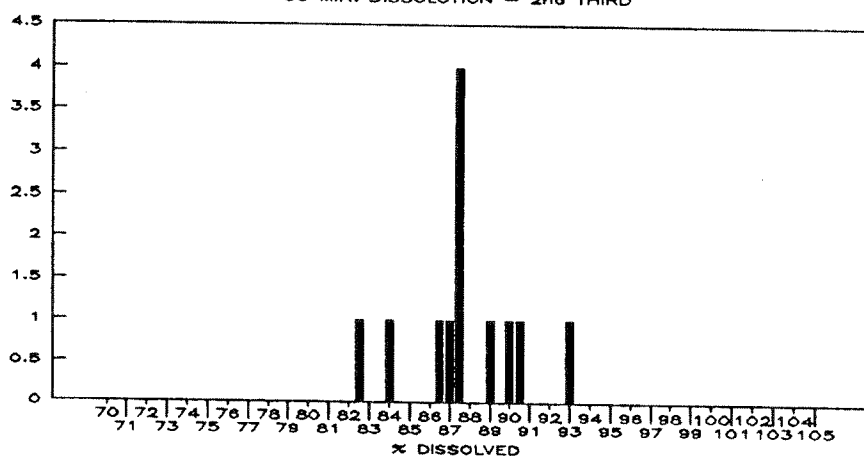
DIGOXIN TABLETS, 0.5 mg — BATCH 4301A
15 MIN. DISSOLUTION — FINAL THIRD



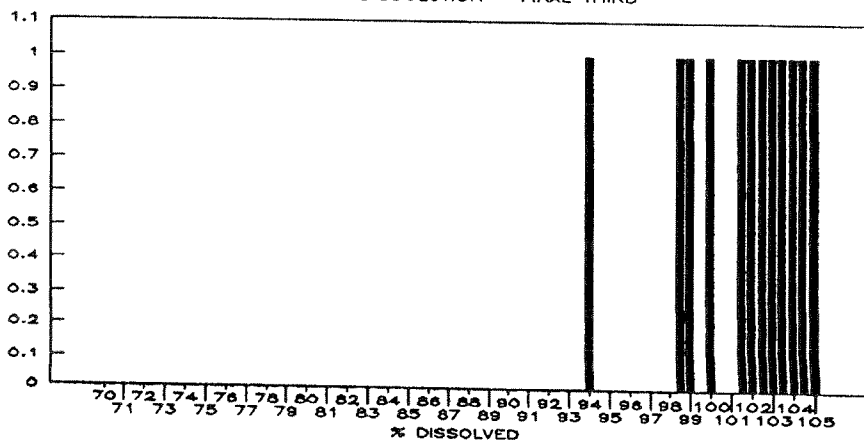
DIGOXIN TABLETS, 0.5 mg — BATCH 4296A
60 MIN. DISSOLUTION — 1st THIRD



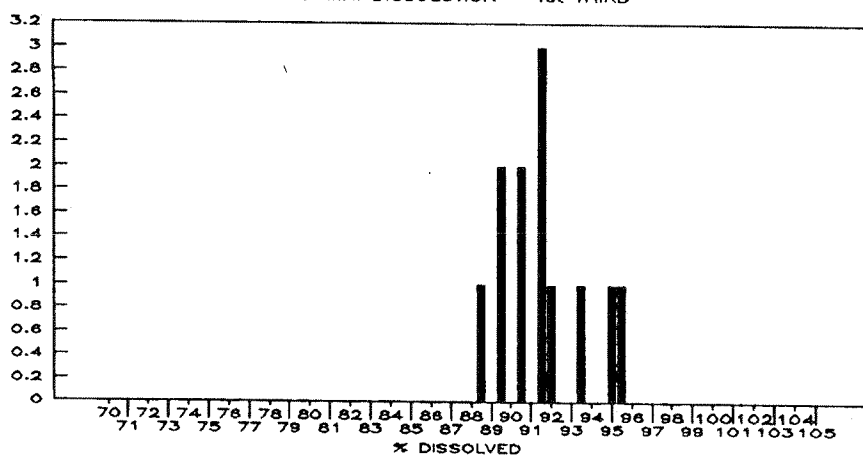
DIGOXIN TABLETS, 0.5 mg — BATCH 4296A
60 MIN. DISSOLUTION — 2nd THIRD



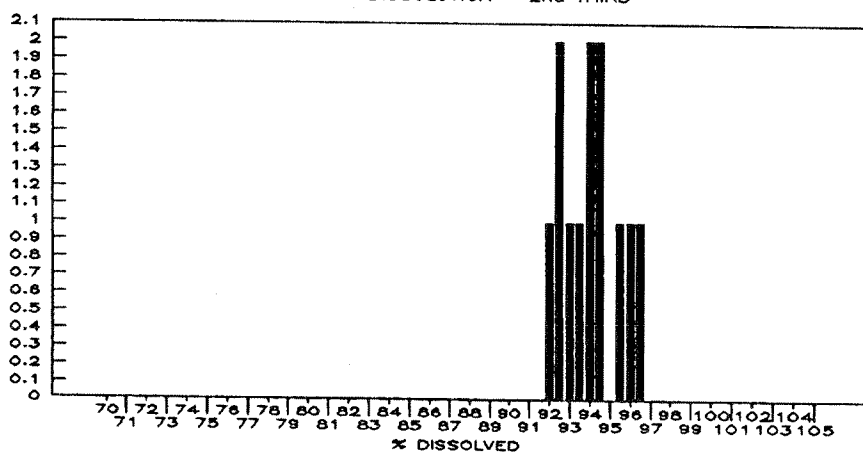
DIGOXIN TABLETS, 0.5 mg — BATCH 4296A
60 MIN. DISSOLUTION — FINAL THIRD



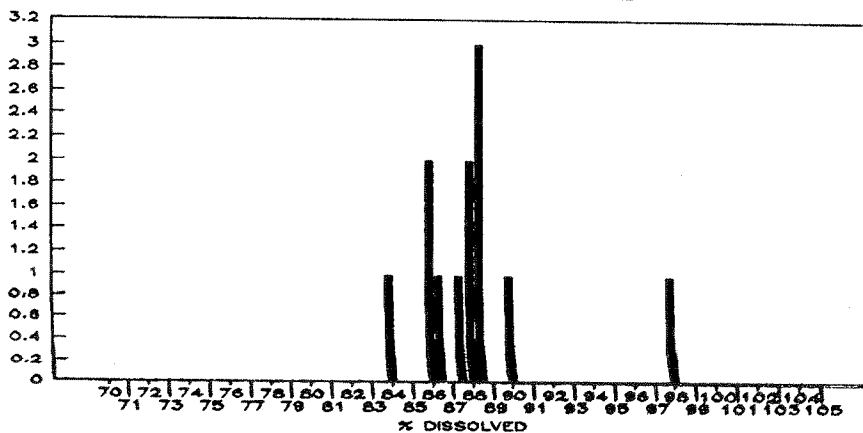
DIGOXIN TABLETS, 0.5 mg — BATCH 4300A
60 MIN. DISSOLUTION — 1st THIRD



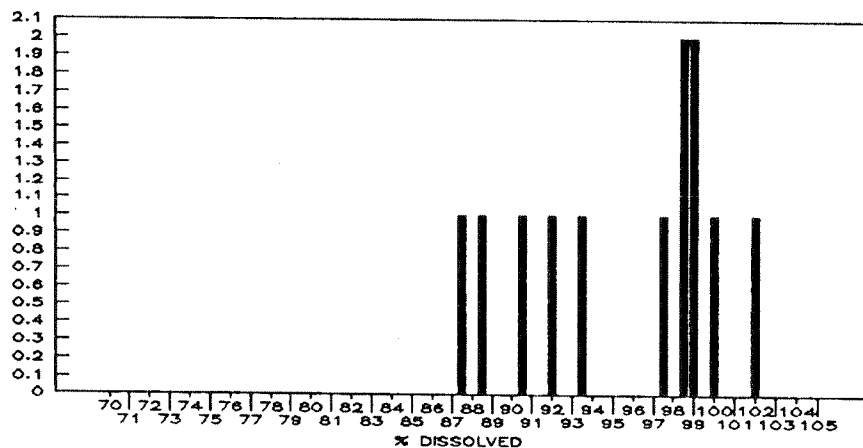
DIGOXIN TABLETS, 0.5 mg — BATCH 4300A
60 MIN. DISSOLUTION — 2nd THIRD



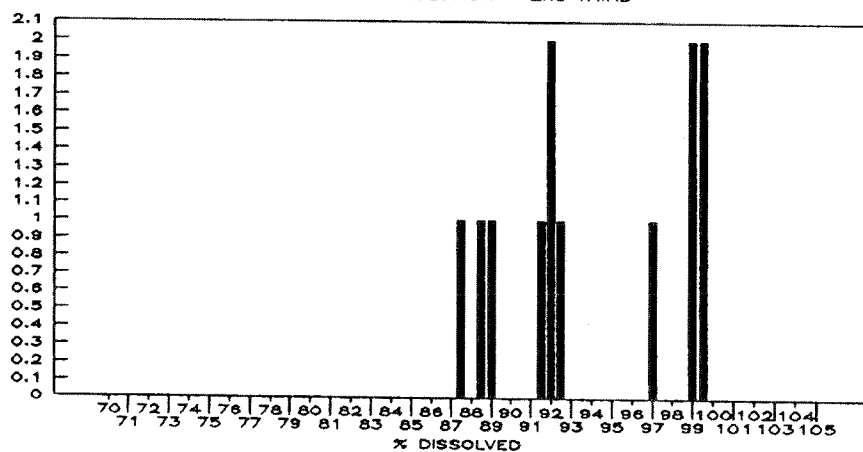
DIGOXIN TABLETS, 0.5 mg — BATCH 4300A
60 MIN. DISSOLUTION — FINAL THIRD



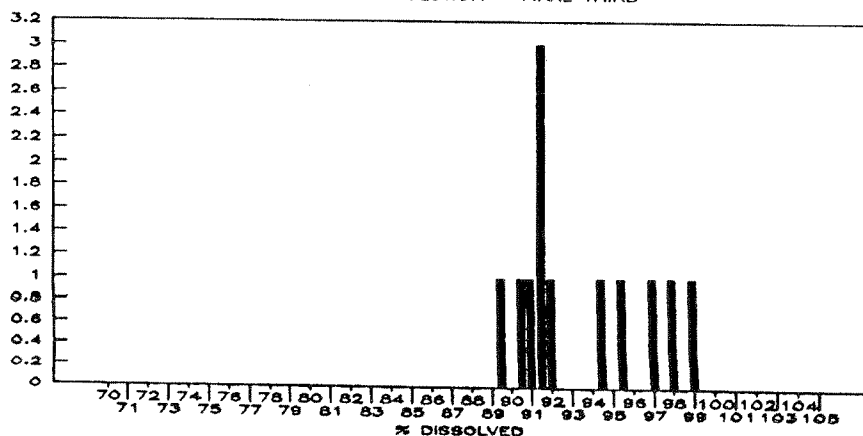
DIGOXIN TABLETS, 0.5 mg — BATCH 4301A
60 MIN. DISSOLUTION — 1st THIRD



DIGOXIN TABLETS, 0.5 mg — BATCH 4301A
60 MIN. DISSOLUTION — 2nd THIRD



DIGOXIN TABLETS, 0.5 mg — BATCH 4301A
60 MIN. DISSOLUTION — FINAL THIRD



AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg**

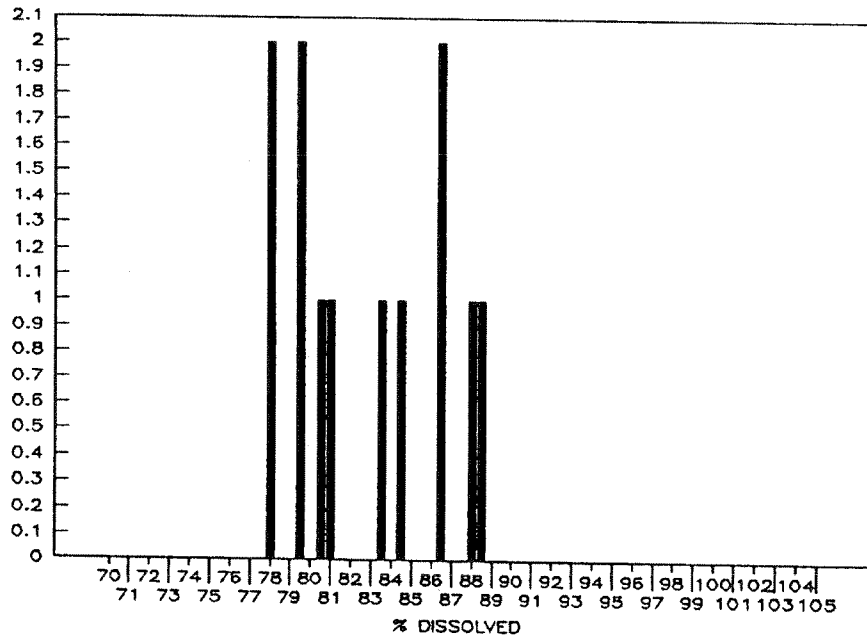
High/Low kp - Dissolution (%) - 15 min.

Batch #	4296A	4296A	4296A	4296A	4300A	4300A	4300A	4300A	4300A	4301A	4301A	4301A	4301A
Sample	High kp	High kp	Low kp	Low kp	High kp	High kp	Low kp	Low kp	High kp	High kp	High kp	Low kp	Low kp
Side	Front	Rear	Front	Rear	Front	Rear	Front	Rear	Front	Rear	Front	Rear	Rear
1	77.9	86.5	75.7	77.6	80.7	80.0	83.0	82.2	77.8	78.5	83.3	85.9	
2	79.1	88.1	75.8	82.0	82.2	82.4	75.7	76.8	81.2	81.9	82.8	83.6	
3	80.1	86.2	75.9	80.2	81.6	82.9	82.1	77.3	78.3	83.2	83.2	83.4	
4	79.5	83.5	76.2	81.5	81.6	83.6	78.7	78.0	81.3	79.8	81.4	85.1	
5	80.7	87.7	76.4	78.9	83.2	80.8	78.7	81.2	81.3	83.9	83.2	84.9	
6	78.0	84.4	77.1	81.5	85.0	82.3	80.8	75.2	83.6	84.3	86.6	79.7	
Average	79.2	86.1	76.2	80.3	82.4	82.0	79.8	78.5	80.6	81.9	83.4	83.8	
St Dev.	1.1	1.8	0.5	1.7	1.5	1.3	2.7	2.7	2.2	2.3	1.7	2.2	
RSD	1.4	2.1	0.7	2.2	1.8	1.6	3.3	3.4	2.7	2.9	2.1	2.6	

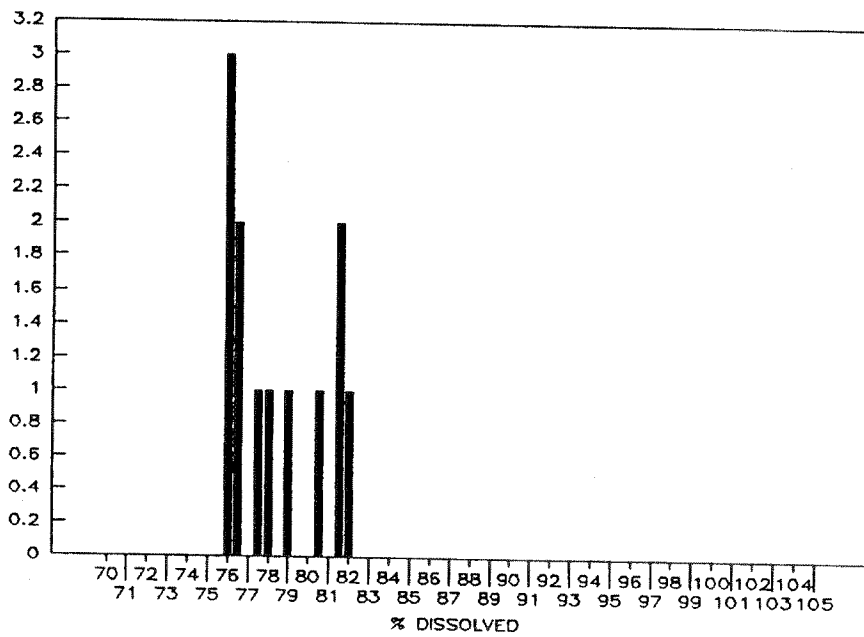
High/Low kp - Dissolution (%) - 60 min.

Batch #	4296A	4296A	4296A	4296A	4300A	4300A	4300A	4300A	4300A	4301A	4301A	4301A	4301A
Sample	High kp	High kp	Low kp	Low kp	High kp	High kp	Low kp	Low kp	High kp	High kp	High kp	Low kp	Low kp
Side	Front	Rear	Front	Rear	Front	Rear	Front	Rear	Front	Rear	Front	Rear	Rear
1	94.2	96.6	92.4	93.9	96.3	99.1	88.6	91.5	97.6	96.4	93.0	99.3	
2	100.5	99.0	96.1	97.9	100.1	98.0	85.9	88.7	98.8	94.6	95.5	101.7	
3	98.1	96.1	94.6	96.0	97.9	99.5	90.8	86.6	96.8	98.4	94.9	98.1	
4	97.3	90.6	92.9	93.7	98.8	100.8	89.6	94.3	99.5	95.3	94.1	100.9	
5	98.8	93.2	97.5	92.7	98.0	98.7	93.6	100.5	97.8	98.9	95.8	100.4	
6	99.7	91.1	93.8	95.3	99.4	98.6	93.6	93.2	97.6	99.2	101.4	100.0	
Average	98.1	94.4	94.6	94.9	98.4	99.1	90.4	92.5	98.0	97.1	95.8	100.1	
St Dev.	2.2	3.3	2.0	1.9	1.3	1.0	3.0	4.9	1.0	2.0	2.9	1.3	
RSD	2.3	3.5	2.1	2.0	1.4	1.0	3.3	5.3	1.0	2.0	3.1	1.3	

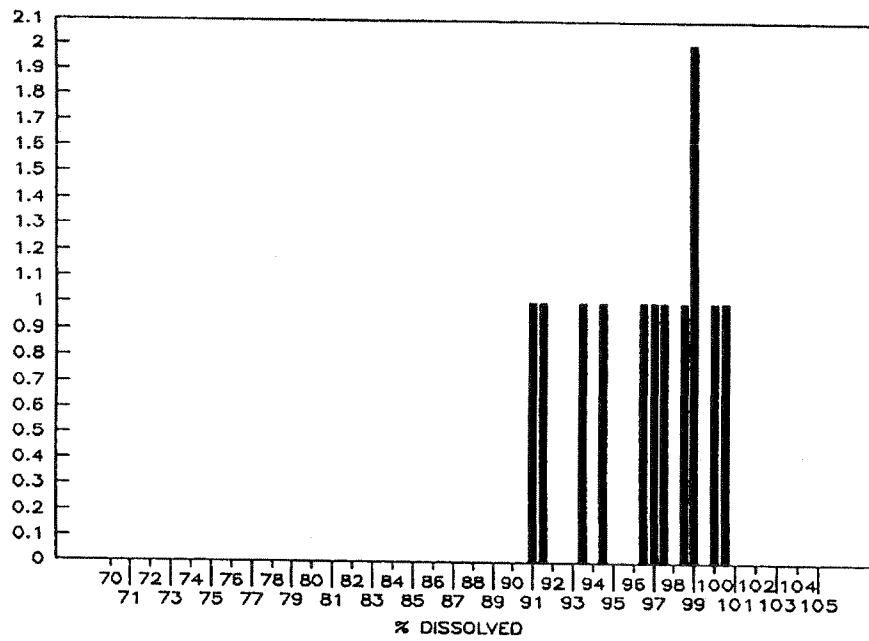
DIGOXIN TABLETS, 0.5 mg — BATCH 4296A
15 MIN. DISSOLUTION — HIGH KP



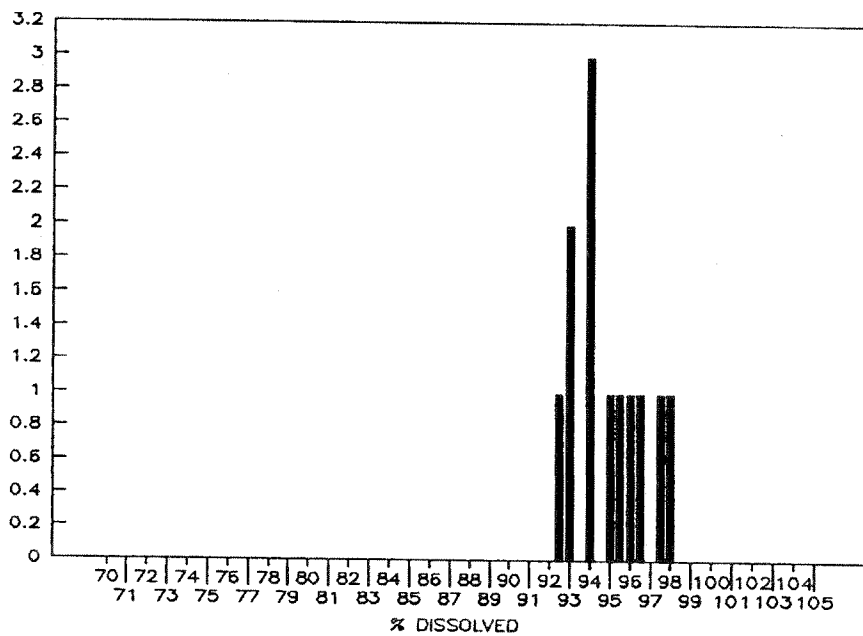
DIGOXIN TABLETS, 0.5 mg — BATCH 4296A
15 MIN. DISSOLUTION — LOW KP



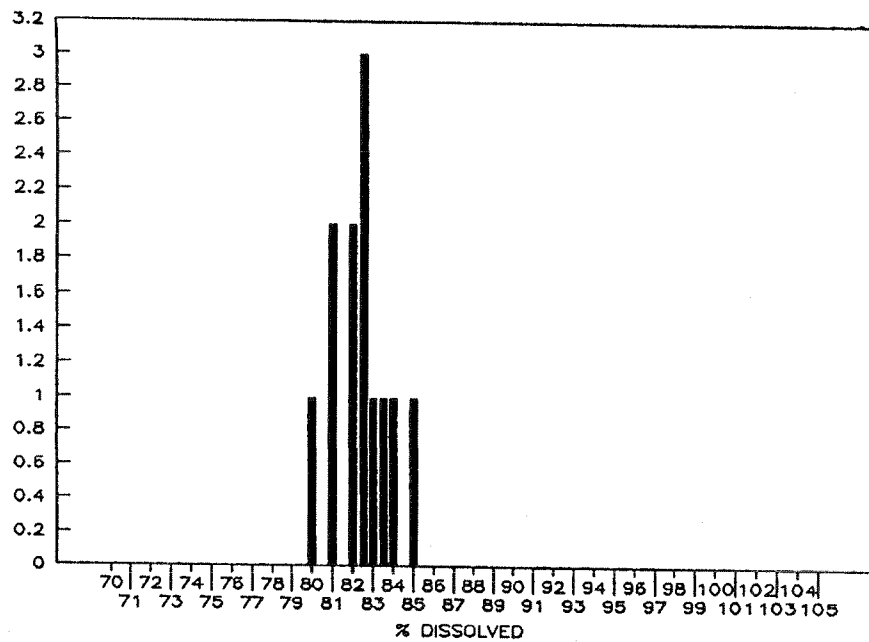
DIGOXIN TABLETS, 0.5 mg — BATCH 4296A
60 MIN. DISSOLUTION — HIGH KP



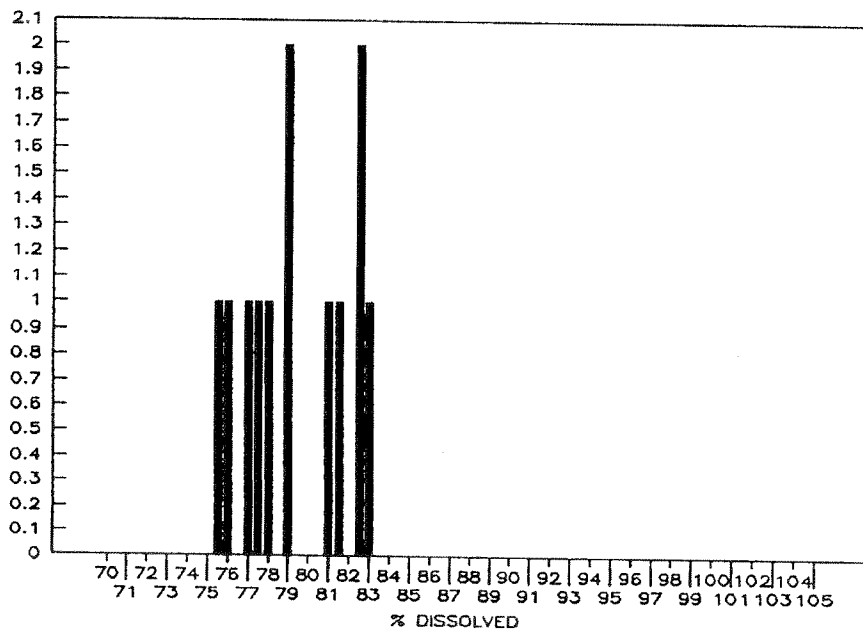
DIGOXIN TABLETS, 0.5 mg — BATCH 4296A
60 MIN. DISSOLUTION — LOW KP



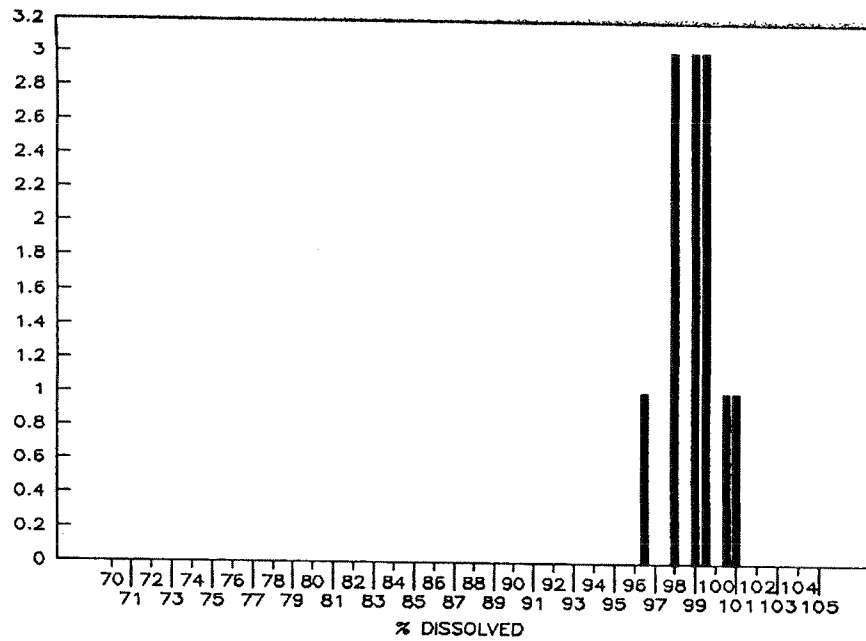
DIGOXIN TABLETS, 0.5 mg — BATCH 4300A
15 MIN. DISSOLUTION — HIGH KP



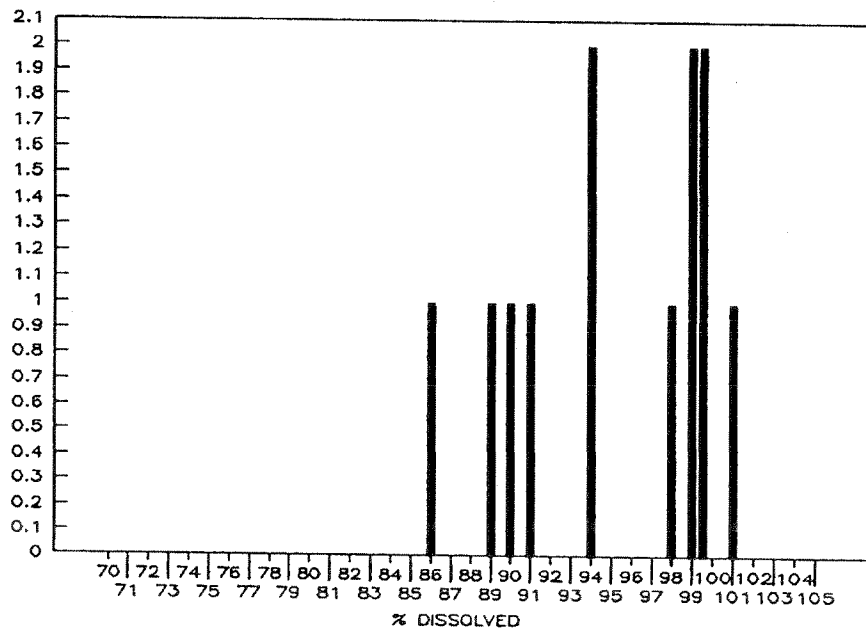
DIGOXIN TABLETS, 0.5 mg — BATCH 4300A
15 MIN. DISSOLUTION — LOW KP



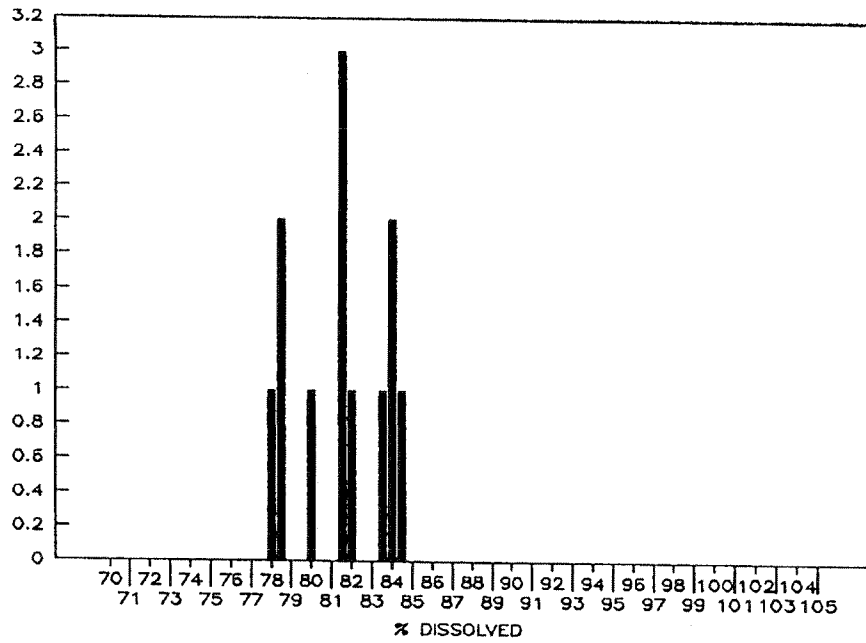
DIGOXIN TABLETS, 0.5 mg – BATCH 4300A
60 MIN. DISSOLUTION – HIGH KP



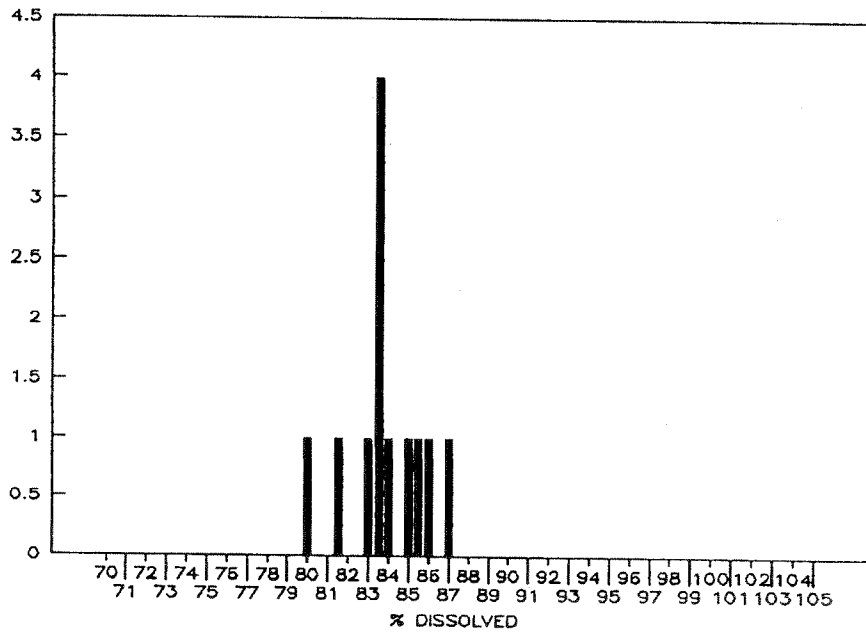
DIGOXIN TABLETS, 0.5 mg – BATCH 4300A
60 MIN. DISSOLUTION – LOW KP



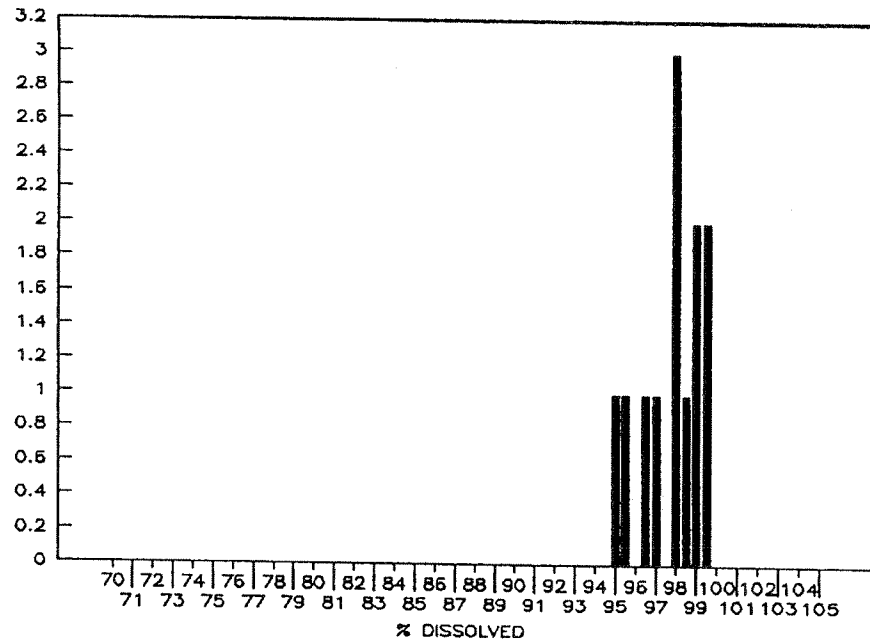
DIGOXIN TABLETS, 0.5 mg – BATCH 4301A
15 MIN. DISSOLUTION – HIGH KP



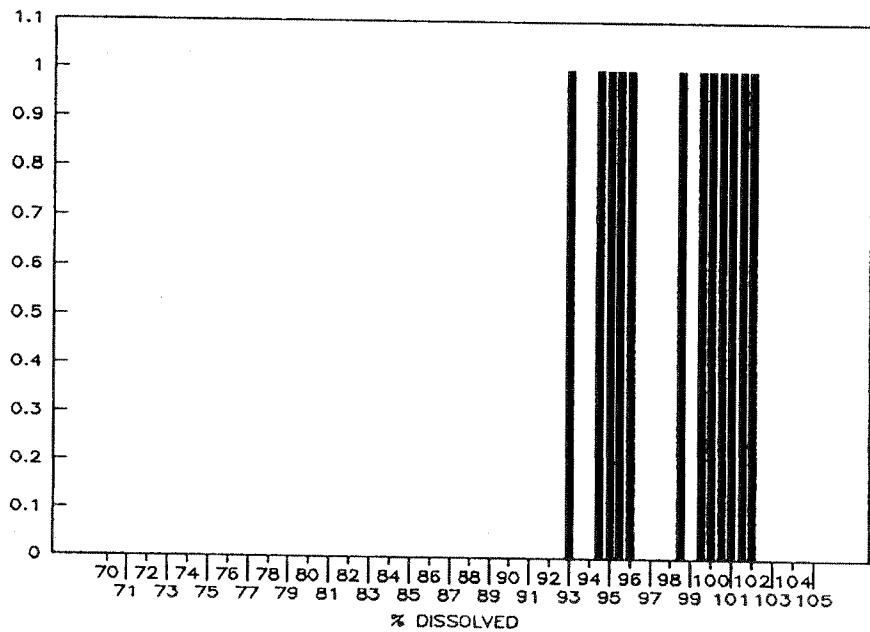
DIGOXIN TABLETS, 0.5 mg – BATCH 4301A
15 MIN. DISSOLUTION – LOW KP



DIGOXIN TABLETS, 0.5 mg — BATCH 4301A
60 MIN. DISSOLUTION — HIGH KP



DIGOXIN TABLETS, 0.5 mg — BATCH 4301A
60 MIN. DISSOLUTION — LOW KP



Amide Pharmaceutical, Inc.

COMPRESSION DEPARTMENT
PROCESS VALIDATION

PRODUCT NAME: Digoxin Tablets 0.5 mgBATCH #: 4296ATABLET PRESS ID #: 66

	Limit	Time
High KP	above 8.0 kp	5.22 pm
Low KP	0.5 - 3.0 kp	5.44 pm
Maximum KP	Not possible	—
Regular Speed	22 rpm	—

	RPM	Time
High Speed	27	7.23 am
Low Speed	17	8.13 am

Done By: <u>Am</u>	Date: <u>10/18/94</u>
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FD301.1

Amide Pharmaceutical, Inc.

Process Validation

Page 1 of 1

COMPRESSION DATA SHEET

Prod Id: 147 Prod Name: Digoxin Tablets 0.5 mg

Batch #: 4296A

Date: 10/18/94

Tablet Press Id: 66 Hardness Tester Id: 255

Thickness Gauge Id: 645 Scale Id: 226

Target Weight (1 Tablet) : 120.0 mg
 Target Weight (10 Tablets) : 1.200 g
 Weight Range (10 Tablets) : 1.274 - 1.326 g

Thickness Limits : 3.0 - 4.0 mm
 Hardness Limits : ~~2.0 - 3.0 KP~~ 1512 10/13/94

High KP - Above 8.0 RPM-22

Front Exit Chute				
Time	Weight (g)	Thickness mm	Hardness KP	Init
5-21 min	1.324	3.28	3.30	7.4
1 min.				6.5
5-25 min	1.326	3.35	3.30	6.9
5 min.				6.4
				6.8
				Am

Rear Exit Chute				
Time	Weight (g)	Thickness mm	Hardness KP	Init
1 min.	1.316	3.27	3.40	3.30
5 min.	1.326	3.29	3.51	6.9
				6.4
				6.9
				Am

* Composite Weight of 10 Tablets

Front Exit Chute				
Time	Weight of Each Tablet (mg)			
1 min.	131	133	134	132
5 min.	133	136	133	133
				136
				135
				132
				130
				134
				133

Rear Exit Chute				
Time	Weight of Each Tablet (mg)			
1 min.	132	134	133	131
5 min.	133	132	135	132
				134
				133
				132
				133
				132
				133

Comments: High Hardness & 8.0 kp can not be obtained maximum hardness achieved

Low 7.6 kp. AM 10-18-94

Amide Pharmaceutical, Inc.

Process Validation

Page 1 of 1

COMPRESSION DATA SHEET

Prod Id: 147 Prod Name: Digoxin Tablets 0.5 mg

Batch #: 42964

Date: 10/15/94

Tablet Press Id: 66 Hardness Tester Id: 255

Thickness Gauge Id: 645 Scale Id: 226

Target Weight (1 Tablet) : 130.0 mg
 Target Weight (10 Tablets) : 1.300 g
 Weight Range (10 Tablets) : 1.274 - 1.326 g

Thickness Limits : 3.0 - 4.0 mm
 Hardness Limits : ~~2.0 - 3.0 KP~~ ^{KSP}

10/13/94

New KP — 0.5 - 3.0 RPM - 22

Front Exit Chute								
Time	Weight (g)	Thickness mm		Hardness KP	Init			
5.46 hr 1 min.	1.291	3.65	3.67	3.64	1.2	1.0	1.5	Am
5.52 hr 5 min.	1.295	3.62	3.64	3.64	1.5	1.1	1.6	Am

Rear Exit Chute						
Time	Weight (g)	Thickness mm	Hardness KP	Init		
1 min.	1.300	3.64	3.64	1.4	1.3	1.2
5 min.	1.303	3.64	3.64	1.1	1.1	1.1

* Composite Weight of 10 Tablets

Front Exit Chute										
Time	Weight of Each Tablet(mg)									
1 min.	131	131	127	132	127	128	129	131	127	128
5 min.	128	129	130	126	129	129	124	130	128	128

Rear Exit Chute									
Time	Weight of Each Tablet(mg)								
1 Min.	131	131	132	129	132	128	130	129	127
5 Min.	131	132	128	130	130	130	132	130	131

Comments:

0187.13

Amide Pharmaceutical, Inc.

Process Validation

Page 1 of 1COMPRESSION DATA SHEET

Prod Id: 147 Prod Name: Digoxin Tablets 0.5 mg

Batch #: 4296A

Date: 10/17/94

Tablet Press Id: 66 Hardness Tester Id: 255

Thickness Gauge Id: 645 Scale Id: 226

Target Weight (1 Tablet) : 130.0 mg
 Target Weight (10 Tablets) : 1.300 g
 Weight Range (10 Tablets) : 1.274 - 1.326 g

Thickness Limits : 3.0 - 4.0 mm
 Hardness Limits : 2.0 - 8.0 KP

High Speed — 27

Front Exit Chute								
Time	Weight* (g)	Thickness mm		Hardness KP		Init		
7.22 hrs 1 min.	1.248	3.34	3.32	3.36	2.7	4.1	3.7	Am
7.26 hrs 5 min.	1.251	3.33	3.30	3.35	2.4	3.4	3.7	Am

Rear Exit Chute								
Time	Weight* (g)	Thickness mm			Hardness Kp		Init	
1 Min.	1.271	3.31	3.38	3.31	3.6	2.3	2.6	Am
5 Min.	1.267	3.34	3.49	3.31	3.4	6.2	3.0	Am

* Composite Weight of 10 Tablets

Front Exit Chute									
Time	Weight of Each Tablet(mg)								
1 Min.	127	127	129	127	122	126	124	121	121
5 Min.	127	125	124	130	134	127	128	133	128

Rear Exit Chute									
Time	Weight of Each Tablet(mg)								
1 Min.	135	135	126	130	132	131	132	131	131
5 Min.	130	136	131	124	125	130	124	121	130

Comments:

Amide Pharmaceutical, Inc.

Process Validation

Page 1 of 1

COMPRESSION DATA SHEET

Prod Id: 147 Prod Name: Digoxin Tablets 0.5 mg

Batch #: 4296A

Date: 10/17/04

Tablet Press Id: 66 Hardness Tester Id: 355

Thickness Gauge Id: 645 Scale Id: 236

Target Weight (1 Tablet) : 130.0 mg
 Target Weight (10 Tablets) : 1.300 g
 Weight Range (10 Tablets) : 1.274 - 1.326 g

Thickness Limits : 3.0 - 4.0 mm
 Hardness Limits : 2.0 - 8.0 KP

slow speed — 17

Front Exit Chute								
Time	Weight (g)	Thickness mm	Hardness KP	Init				
8.12 min	1.296	3.33	3.35	3.57	4.2	4.2	4.5	4.1
1 Min.								
8.16 min	1.308	3.37	3.35	3.36	4.5	4.5	4.5	4.1
5 Min.								

Rear Exit Chute					
Time	Weight (g)	Thickness mm	Hardness KP	Init	
1 Min.	1.310	3.33	3.35	3.35	4.6
5 Min.	1.307	3.36	3.33	3.6	4.6

* Composite Weight of 10 Tablets

Front Exit Chute										
Time	Weight of Each Tablet(mg)									
1 Min.	130	132	127	132	132	133	128	132	133	131
5 Min.	129	132	133	134	130	128	132	132	131	131

Rear Exit Chute										
Time	Weight of Each Tablet(mg)									
1 Min.	131	131	130	131	130	130	131	132	131	129
5 Min.	133	129	132	130	130	131	129	130	131	130

Comments:

Amide Pharmaceutical, Inc.

COMPRESSION DEPARTMENT

PROCESS VALIDATION

PRODUCT NAME: Digoxin Tablets 0.5mg (147)BATCH #: 4300ATABLET PRESS ID #: 66

	Limit	Time
High KP	above 8.0 /cp	7.51 Am
Low KP	0.5 - 3.0 /cp	8.08 Am
Maximum KP	not possible	—
Regular Speed	22 rpm	—

	RPM	Time
High Speed	27	8.25 Am
Low Speed	17	8.40 Am

Done By: <u>K. B</u>	Date: <u>10/21/94</u>
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Amide Pharmaceutical, Inc.

Process Validation

Page 1 of 1

COMPRESSION DATA SHEET

Prod Id: 147 Prod Name: Digoxin Tablets 0.5 mg

Batch #: 4200A

Date: 10/21/94

Tablet Press Id: 66

Hardness Tester Id: 254

Thickness Gauge Id: 643

Scale Id: 217

Target Weight (1 Tablet) : 130.0 mg
 Target Weight (10 Tablets) : 1.300 g
 Weight Range (10 Tablets) : 1.274 - 1.326 g

Thickness Limits : 3.0 - 4.0 mm
 Hardness Limits : ~~2.0~~ 8.0 KP

High KP — Above 8.0 KP RPM 22

10/18/94

Front Exit Chute				
Time	Weight (g)	Thickness mm	Hardness KP	Init
7:50 AM	1.286	3.25	3.24	3.26
1 min.				
7:54 AM	1.289	3.23	3.25	3.27
5 min.				

Rear Exit Chute				
Time	Weight (g)	Thickness mm	Hardness KP	Init
1 min.	1.266	3.23	3.25	3.23
5 min.	1.273	3.23	3.23	3.24

* Composite Weight of 10 Tablets

Front Exit Chute				
Time	Weight of Each Tablet (mg)			
1 min.	128	135	130	139
5 min.	128	127	129	125

Rear Exit Chute				
Time	Weight of Each Tablet (mg)			
1 min.	125	129	128	126
5 min.	126	127	127	127

Comments: High KP hardness of more than 8 KP can not be obtained highest value obtained was 7.2 KP. AN 10-21-94

<P0187.1>

Amide Pharmaceutical, Inc.

Process Validation

Page 1 of 1COMPRESSION DATA SHEETProd Id: 147 Prod Name: Digoxin Tablets 0.5 mgBatch #: 4300ADate: 10/21/99Tablet Press Id: 66 Hardness Tester Id: 254Thickness Gauge Id: 643 Scale Id: 217

Target Weight (1 Tablet) : 130.0 mg
 Target Weight (10 Tablets) : 1.300 g
 Weight Range (10 Tablets) : 1.274 - 1.326 g

Thickness Limits : 3.0 - 4.0 mm
 Hardness Limits : ~~2.0 - 8.0 KP~~ 10/18/99

Low KP 0.5 - 3.0 RPN 22

Front Exit Chute							
Time	Weight* (g)	Thickness mm	Hardness KP		Init		
8, 0.7 min 1 Min.	1.266	3.62	3.63	0.9	0.9	0.8	Am
9.11 min 5 Min.	1.273	3.62	3.63	0.9	1.2	0.7	Am

Rear Exit Chute								
Time	Weight* (g)	Thickness mm			Hardness KP		Init	
1 Min.	1.249	3.61	3.62	3.61	0.8	0.7	0.9	Am
5 Min.	1.250	3.60	3.60	3.62	1.0	0.9	0.8	Am

* Composite Weight of 10 Tablets

Front Exit Chute									
Time	Weight of Each Tablet(mg)								
1 Min.	127	122	127	125	128	132	125	124	128
5 Min.	127	124	125	124	126	127	125	128	126

Rear Exit Chute									
Time	Weight of Each Tablet(mg)								
1 Min.	123	124	127	128	124	125	123	126	127
5 Min.	124	125	124	125	127	126	126	123	124

Comments:

Amide Pharmaceutical, Inc.

Process Validation

Page 1 of 1

COMPRESSION DATA SHEET

Prod Id: 147 Prod Name: Digoxin Tablets 0.5 mg

Batch #: 4300A

Date: 10/21/94

Tablet Press Id: 66 Hardness Tester Id: 254

Thickness Gauge Id: 643 Scale Id: 217

Target Weight (1 Tablet) : 130.0 mg
 Target Weight (10 Tablets) : 1.300 g
 Weight Range (10 Tablets) : 1.274 - 1.326 g

Thickness Limits : 3.0 - 4.0 mm
 Hardness Limits : 2.0 - 8.0 KP

High Speed — RPM. 27

Front Exit Chute								
Time	Weight (g)	Thickness mm	Hardness KP	Init				
3:24 AM 1 Min.	1.204	3.09	3.27	3.13	1.6	3.8	1.8	Am
3:28 AM 5 Min.	1.147	3.20	3.13	3.18	1.7	2.5	2.3	Am

Rear Exit Chute						
Time	Weight* (g)	Thickness mm	Hardness KP	Init		
1 Min.	1.210	3.22 3.27 3.23	4.4 2.4 3.3	Am		
5 Min.	1.218	3.26 3.21 3.32	3.5 2.8 2.7	Am		

* Composite Weight of 10 Tablets

Front Exit Chute											
Time		Weight of Each Tablet(mg)									
1 Min.		111	113	107	107	113	121	113	128	117	121
5 Min.		118	117	117	121	125	125	114	125	117	126

Rear Exit Chute										
Time	Weight of Each Tablet(mg)									
1 Min.	130	126	124	120	125	125	124	115	122	124
5 Min.	119	120	118	121	119	124	120	125	123	124

Comments:

<P0187.1>

Amide Pharmaceutical, Inc.

Process Validation

Page 1 of 1

COMPRESSION DATA SHEET

Prod Id: 147 Prod Name: Digoxin Tablets 0.5 mg

Batch #: 4300A

Date: 10/21/94

Tablet Press Id: 66 Hardness Tester Id: 254

Thickness Gauge Id: 643 Scale Id: 217

Target Weight (1 Tablet) : 130.0 mg
 Target Weight (10 Tablets) : 1.300 g
 Weight Range (10 Tablets) : 1.274 - 1.326 g

Thickness Limits : 3.0 - 4.0 mm
 Hardness Limits : 2.0 - 8.0 KP

Two Speed - RPM 17

Front Exit Chute								
Time	Weight (g)	Thickness mm	Hardness KP	Init				
(P-30 AM) 1 Min.	1.277	3.32	3.32	3.49	4.2	4.3	4.2	Am
5 Min.	1.279	3.32	3.39	3.30	4.0	4.1	3.9	Am

Rear Exit Chute								
Time	Weight (g)	Thickness mm	Hardness Kp	Init				
1 Min.	1.291	3.30	3.34	3.31	4.3	4.2	4.3	Am
5 Min.	1.294	3.29	3.33	3.35	4.3	4.1	3.9	Am

* Composite Weight of 10 Tablets

Front Exit Chute										
Time	Weight of Each Tablet(mg)									
1 min.	127	129	131	127	125	127	129	126	127	129
5 min.	128	130	128	129	130	126	120	128	128	124

Rear Exit Chute										
Time	Weight of Each Tablet(mg)									
1 Min.	130	132	128	130	130	128	129	130	131	129
5 Min.	129	130	132	129	127	130	128	131	130	130

Comments:

<P0187.1>

Amide Pharmaceutical, Inc.

COMPRESSION DEPARTMENT**PROCESS VALIDATION**PRODUCT NAME: Digitoxin Tablets 0.5 mg (147)BATCH #: 4301 ATABLET PRESS ID #: 66

	Limit	Time
High KP	above 8.0 /cp	1.45 per
Low KP	0.5 - 3.0 /cp	2.05 per
Maximum KP	Not Possible	—
Regular Speed	22	—

	RPM	Time
High Speed	28	2.28 per
Low Speed	17	3.00 per

Done By: <u>Am</u>	Date: <u>10/21/94</u>
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PD301.1

Amide Pharmaceutical, Inc.

Process Validation

Page 1 of 1

COMPRESSION DATA SHEET

Prod Id: 147 Prod Name: Digoxin Tablets 0.5 mg

Batch #: 4301A

Date: 10/11/94

Tablet Press Id: 66 Hardness Tester Id: 354

Thickness Gauge Id: 643 Scale Id: 317

Target Weight (1 Tablet) : 130.0 mg
 Target Weight (10 Tablets) : 1.300 g
 Weight Range (10 Tablets) : 1.274 - 1.326 g

Thickness Limits : 3.0 - 4.0 mm
 Hardness Limits : ~~2.0~~ ~~8.0~~ ~~KP~~ ~~KSP~~ 10/18/94

High KP — Above 8.0 KP RPM 22

Front Exit Chute				
Time	Weight* (g)	Thickness mm	Hardness KP	Init
1-44 PM	1.331	3.33	3.32	3.33
1 Min.		6.1	6.7	6.0
5 Min.	1.339	3.32	3.36	3.34
		5.8	5.8	6.7
				PM

* Composite Weight of 10 Tablets

Rear Exit Chute				
Time	Weight* (g)	Thickness mm	Hardness KP	Init
1 Min.	1.329	3.32	3.30	3.32
		7.0	6.0	6.6
5 Min.	1.319	3.33	3.28	3.29
		7.7	7.4	7.0
				PM

Front Exit Chute				
Time	Weight of Each Tablet (mg)			
1 Min.	133	134	133	135
		135	135	136
			130	135
				132
				133
5 Min.	134	133	135	171
			134	135
			136	134
				141
				138

Rear Exit Chute				
Time	Weight of Each Tablet (mg)			
1 Min.	131	131	132	133
		133	131	130
			132	142
				133
				130
5 Min.	131	134	132	133
			133	132
			132	132
				133
				132

Comments: High KP hardness at 8.0 KP cannot be obtained, highest hardness observed was 7.4 KP at 10-21-94.

<P0187.1>

Amide Pharmaceutical, Inc.

Process Validation

Page 1 of 1

COMPRESSION DATA SHEET

Prod Id: 147 Prod Name: Digoxin Tablets 0.5 mg

Batch #: 4301A

Date: 10/21/94

Tablet Press Id: 66 Hardness Tester Id: 254

Thickness Gauge Id: 643 Scale Id: 217

Target Weight (1 Tablet) : 130.0 mg
 Target Weight (10 Tablets) : 1.300 g
 Weight Range (10 Tablets) : 1.274 - 1.326 g

Thickness Limits : 3.0 - 4.0 mm
 Hardness Limits : 2.0 - 8.0 KSP 10/13/94

done KP - 0.5 to 3.0 RSPM 22

Front Exit Chute						
Time	Weight (g)	Thickness mm	Hardness KP	Init		
2.04 min	1.307	3.63	3.64	1.4	1.7	1.6
1 min.						
2.05 min	1.301	3.60	3.63	3.62	1.4	1.8
5 min.						

* Composite Weight of 10 Tablets

Front Exit Chute										
Time	Weight of Each Tablet(mg)									
1 min.	129	132	130	131	133	128	130	130	130	129
5 min.	128	133	129	131	128	132	131	130	128	129

Rear Exit Chute									
Time	Weight* (g)	Thickness mm			Hardness KP			Init	
1 Min.	1.309	3.61	3.63	3.60	1.6	1.8	2.7	A1	
5 Min.	1.305	3.59	3.58	3.61	1.6	1.6	1.4	A1	

Rear Exit Chute										
Time	Weight of Each Tablet(mg)									
1 Min.	130	130	127	134	130	131	130	130	130	131
5 Min.	134	130	132	133	130	132	130	130	131	134

Comments:

<P0187.1>

Amide Pharmaceutical, Inc.

Process Validation

Page 1 of 1

COMPRESSION DATA SHEET

Prod Id: 147 Prod Name: Digoxin Tablets 0.5 mg

Batch #: 4301A

Date: 10/21/94

Tablet Press Id: 66

Hardness Tester Id: 284

Thickness Gauge Id: 643

Scale Id: 217

Target Weight (1 Tablet) : 130.0 mg
 Target Weight (10 Tablets) : 1.300 g
 Target Weight Range (10 Tablets) : 1.274 - 1.326 g

Thickness Limits : 3.0 - 4.0 mm
 Hardness Limits : 2.0 - 8.0 KP

11/16/94 High Speed — RPM 28

Front Exit Chute				
Time	Weight (g)	Thickness mm	Hardness KP	Init
1 Min.	1.321	3.35	5.1	5.6
5 Min.	1.314	3.36	5.4	5.2

Rear Exit Chute				
Time	Weight (g)	Thickness mm	Hardness KP	Init
1 Min.	1.325	3.34	5.3	5.4
5 Min.	1.328	3.35	5.4	5.2

* Composite Weight of 10 Tablets

Front Exit Chute									
Time	Weight of Each Tablet (mg)								
1 Min.	133	131	130	133	132	132	131	132	133
5 Min.	134	134	132	131	130	131	131	130	132

Rear Exit Chute									
Time	Weight of Each Tablet (mg)								
1 Min.	132	134	132	132	133	134	133	134	133
5 Min.	131	134	134	133	131	135	133	134	132

Comments:

<P0187.1>

Amide Pharmaceutical, Inc.

Process Validation

Page 1 of 1

COMPRESSION DATA SHEET

Prod Id: 147 Prod Name: Digoxin Tablets 0.5 mg

Batch #: 4301A

Date: 10/21/94

Tablet Press Id: 66 Hardness Tester Id: 255

Thickness Gauge Id: 645 Scale Id: 226

Target Weight (1 Tablet) : 130.0 mg
 Target Weight (10 Tablets) : 1.300 g
 Weight Range (10 Tablets) : 1.274 - 1.326 g

Thickness Limits : 3.0 - 4.0 mm
 Hardness Limits : 2.0 - 8.0 KP

Handwritten: 7/11/8/94
 Handwritten: High Speed - 17 RPM

Front Exit Chute									
Time	Weight* (g)	Thickness mm			Hardness KP	Init			
2-56 PM 1 Min.	1.244	3.38	3.35	3.43	3.5	3.5	3.6	3.7	3.9
3-23 PM 5 Min.	1.247	3.37	3.31	3.31	3.5	3.7	3.9	4.1	

Rear Exit Chute									
Time	Weight*	Thickness			Hardness			Init	
	(g)	mm			KP				
1 Min.	1.251	3.42	3.37	3.30	3.8	4.3	4.0	avg	
5 Min.	1.250	3.38	3.34	3.26	3.8	3.7	3.5	avg	

* Composite Weight of 10 Tablets

Front Exit Chute										
Time	Weight of Each Tablet(mg)									
1 Min.	127	129	128	127	135	135	134	136	136	131
5 Min.	121	122	127	120	115	120	121	121	120	116

Rear Exit Chute										
Time	Weight of Each Tablet(mg)									
1 Min.	123	126	126	124	123	125	122	127	126	124
5 Min.	123	123	128	126	129	125	123	126	124	126

Comments:

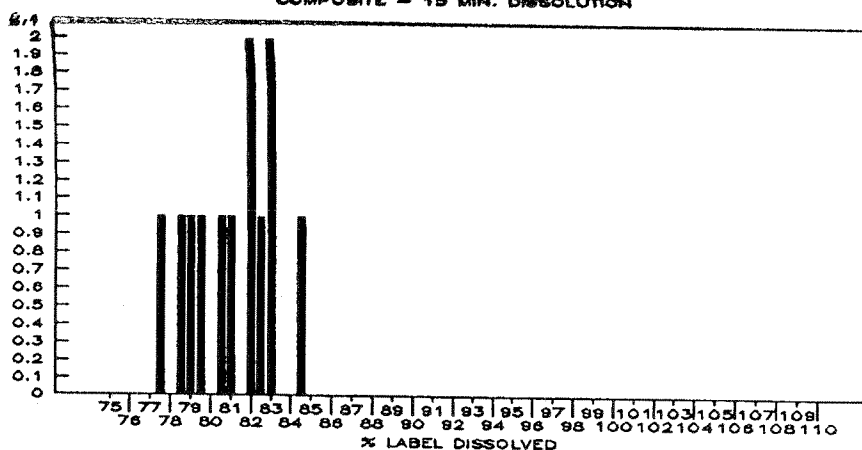
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AMIDE PHARMACEUTICAL, INC.**PROCESS VALIDATION****DIGOXIN TABLETS, 0.5 mg**

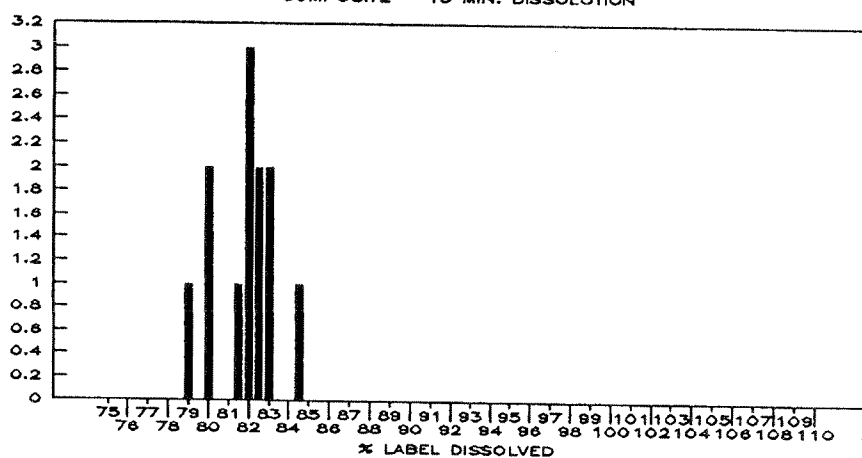
Compression - Composite Dissolution (%)

Batch #	4296A	4296A	4300A	4300A	4301A	4301A
Time	15 min.	60 min.	15 min.	60 min.	15 min.	60 min.
1	84.2	93.0	84.3	96.5	78.7	94.1
2	82.6	91.6	81.7	90.7	77.8	95.7
3	80.1	90.0	81.1	87.2	73.7	92.4
4	80.8	93.1	82.3	88.3	77.5	97.1
5	78.5	92.4	83.0	89.0	78.3	88.7
6	81.8	94.8	81.8	88.9	79.0	88.4
7	79.0	89.8	82.9	90.0	80.6	90.7
8	82.6	93.5	82.0	89.1	77.9	91.0
9	81.9	93.0	82.2	88.3	78.7	91.9
10	79.1	92.2	78.8	88.3	77.0	90.2
11	82.4	99.5	79.7	86.5	75.9	89.0
12	77.1	97.4	79.9	88.5	72.4	89.3
Average	80.8	93.4	81.6	89.3	77.7	91.5
St Dev.	2.1	2.8	1.6	2.5	1.7	2.8
RSD	2.6	3.0	1.9	2.8	2.2	3.1

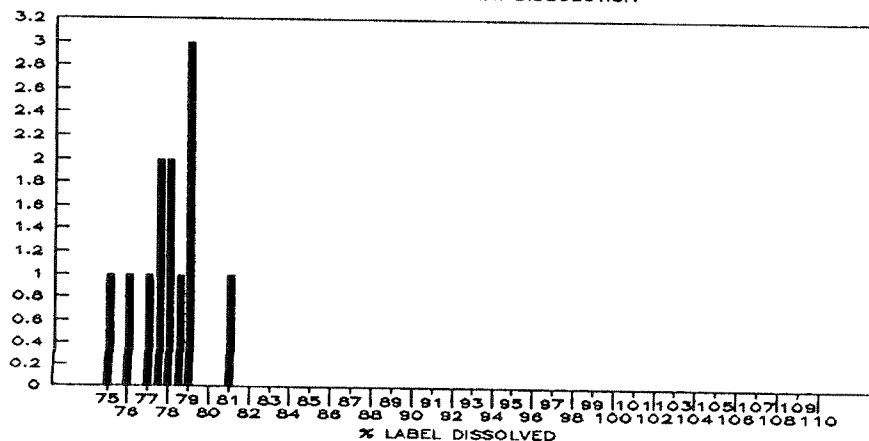
DIGOXIN TABLETS, 0.5 mg — BATCH 4296A
COMPOSITE — 15 MIN. DISSOLUTION



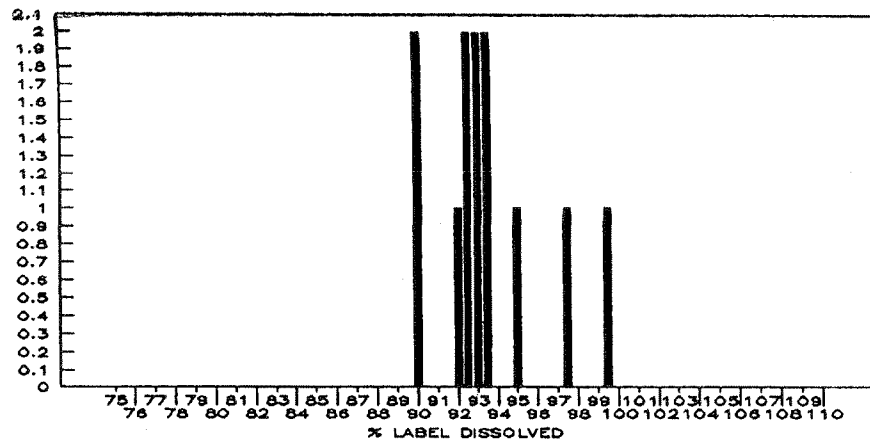
DIGOXIN TABLETS, 0.5 mg — BATCH 4300A
COMPOSITE — 15 MIN. DISSOLUTION



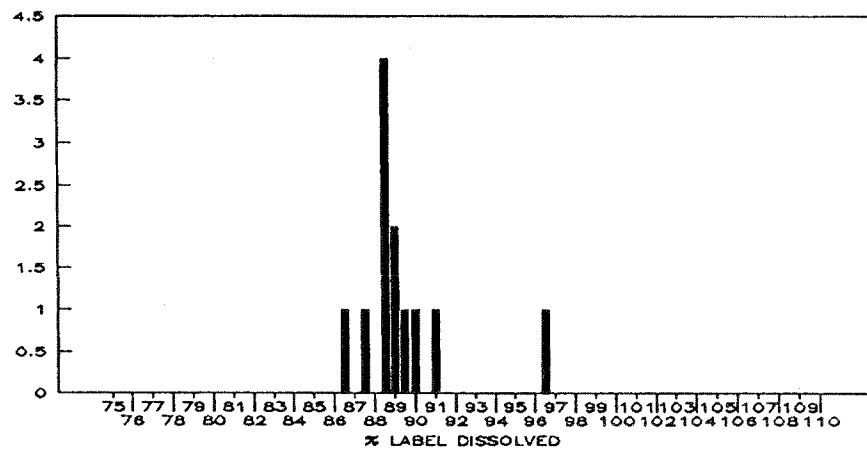
DIGOXIN TABLETS, 0.5 mg — BATCH 4301A
COMPOSITE — 15 MIN. DISSOLUTION



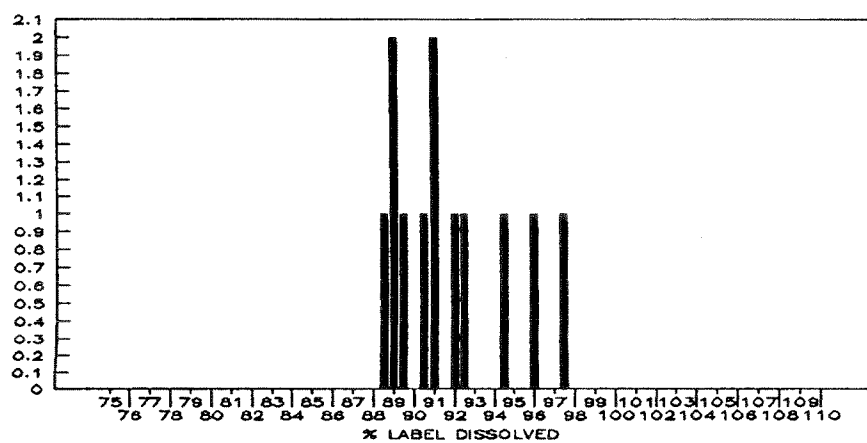
DIGOXIN TABLETS, 0.5 mg — BATCH 4296A
COMPOSITE — 60 MIN. DISSOLUTION



DIGOXIN TABLETS, 0.5 mg — BATCH 4300A
COMPOSITE — 60 MIN. DISSOLUTION



DIGOXIN TABLETS, 0.5 mg — BATCH 4301A
COMPOSITE — 60 MIN. DISSOLUTION

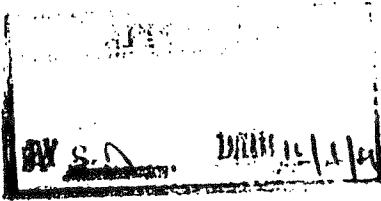


Amide Pharmaceutical, Inc.

Page 1 of 2

LABORATORY TEST REPORT
FINISHED DRUG PRODUCT

PRODUCT: Digoxin Tablets 0.5 mgSPECIFICATION: USPCONTROL #: 4296 ACHEMIST: BR/PK/K.A VOLUME #: 302.02/321.04 PAGE #: 93/107 DATE: 11/1/94SAMPLE STAGE: Overall composite of the batch * Form was filed by KA 11/1/94

TEST	RESULT	LIMIT
DESCRIPTION: Color:	Green	Green
Profile:	Round bisected tablet	Round Bisected Tablets
Other: Debossed	"A" on bisected side of the tablet	"A 147" on bisected side of the tablet
THICKNESS: (Guideline)	3.4 mm	3.0 mm to 4.0 mm
WEIGHT VARIATION:	130.7 mg	± 10% Theo. wt (130 mg) 117.0 mg - 143.0 mg
FRIABILITY:	0.06 %	NMT 1.0 %
IDENTIFICATION: (A)	The R _h time of the major peak in the chromatogram of assay prepn corresponds to Std Prepn	The retention time of the major peak in the chromatogram of assay preparation corresponds to standard preparation.
ASSAY: Digoxin, 0.5 mg	101.3 %	90.0% to 105.0%
UNIFORMITY OF DOSAGE UNITS: (Content Uniformity)	1) 104.3 % 6) 101.2 % 2) 102.1 % 7) 102.7 % 3) 101.9 % 8) 102.6 % 4) 102.9 % 9) 102.0 % 5) 101.9 % 10) 100.7 % AV: 102.2 RSD: 1.0 %	85.0% to 115.0%
		RSD: NMT 6.0%
<input checked="" type="checkbox"/> COMPLIES	PREPARED BY: <u>Nilesh Patel</u>	DATE: <u>11/1/94</u>
<input type="checkbox"/> DOES NOT COMPLY	APPROVED BY: <u>Suryakant Patel</u>	DATE: <u>11/1/94</u>

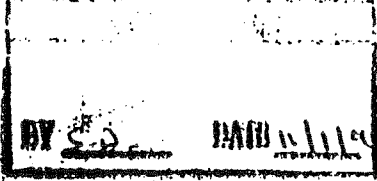
ac13-147c

Pharmaceutical, Inc.

Page 2 of 2

LABORATORY TEST REPORT
FINISHED DRUG PRODUCT

PRODUCT: Digoxin Tablets, 0.5 mgSPECIFICATION: USPCONTROL #: 4296 ACHEMIST: KAVOLUME #: 326.01PAGE #: 131DATE: 11/1/99SAMPLE STAGE: Overall composite of the batch

TEST	RESULT	LIMIT
DISSOLUTION: Media: 500mL 0.1N HCl Appar: I, rpm: 120 Temp: 37°C ± 0.5°C Time: 60 minutes 	15 minutes: 1) <u>84.2</u> % 7) <u>79.0</u> % 2) <u>82.6</u> % 8) <u>82.6</u> % 3) <u>80.1</u> % 9) <u>81.9</u> % 4) <u>80.8</u> % 10) <u>79.1</u> % 5) <u>78.5</u> % 11) <u>82.4</u> % 6) <u>81.8</u> % 12) <u>77.1</u> % Average: <u>80.8</u> / . % 60 minutes: 1) <u>93.0</u> % 7) <u>89.8</u> % 2) <u>91.6</u> % 8) <u>93.5</u> % 3) <u>90.0</u> % 9) <u>93.0</u> % 4) <u>93.1</u> % 10) <u>92.2</u> % 5) <u>92.4</u> % 11) <u>99.5</u> % 6) <u>94.8</u> % 12) <u>97.4</u> % Average: <u>93.3</u> %	(Note - The specified tolerances are for % dissolved, and are not to be interpreted as Q values.) NLT 80% of the LC of Digoxin dissolved in 60 minutes for the average of 12 tablets tested and no individual tablet has less than 75% of the LC of Digoxin dissolved in 60 minutes. If the amount of Digoxin dissolved in 60 minutes is more than 95% for any individual Tablet, the amount dissolved in 15 minutes is not more than 90% for each individual Tablet. (LC: Labeled amount)
<input checked="" type="checkbox"/> COMPLIES <input type="checkbox"/> DOES NOT COMPLY	PREPARED BY: <u>Nilesh Patel</u> APPROVED BY: <u>Suraj Kant Patel</u>	DATE: <u>11/1/99</u> DATE: <u>11/1/99</u>

QC12-147d1

Amide Pharmaceutical, Inc.

Page 1 of 2

LABORATORY TEST REPORTFINISHED DRUG PRODUCTPRODUCT: Digoxin Tablets 0.5 mgSPECIFICATION: USPCONTROL #: 4300 ACHEMIST: PK/SJ VOLUME #: 321.04 / 316.02 PAGE #: 124 / 15 DATE: 11/11/94SAMPLE STAGE: ^{KA} Overall composite of the batch
Report was filed by KA 11/11/94

TEST	RESULT	LIMIT
DESCRIPTION: color:	<u>Green</u>	Green
Profile:	<u>Round bisected tablets</u>	Round Bisected Tablets
Other: Debossed	<u>"A" on bisected side of the tablet</u>	"A 147" on bisected side of the tablet
THICKNESS: (Guideline)	<u>3.4 mm</u>	3.0 mm to 4.0 mm
WEIGHT VARIATION:	<u>130.0 mg</u>	± 10% Theo. wt (130 mg) 117.0 mg - 143.0 mg
FRIABILITY:	<u>0.04 %</u>	NMT 1.0 %
IDENTIFICATION: (A)	<u>The retention time of the major peak in the chromatogram of Assay prep corresponds to the STD Preparation</u>	The retention time of the major peak in the chromatogram of Assay preparation corresponds to standard preparation.
ASSAY: Digoxin, 0.5 mg	<u>100.3 %</u>	90.0% to 105.0%
UNIFORMITY OF DOSAGE UNITS: (Content Uniformity)	1) <u>99.7 %</u> 6) <u>100.2 %</u> 2) <u>100.1 %</u> 7) <u>101.0 %</u> 3) <u>99.3 %</u> 8) <u>101.6 %</u> 4) <u>100.8 %</u> 9) <u>100.6 %</u> 5) <u>101.4 %</u> 10) <u>100.7 %</u> AV: <u>100.5 %</u> RSD: <u>0.8 %</u>	85.0% to 115.0%
<div style="border: 1px solid black; padding: 5px; width: fit-content;"> APPROVED BY <u>SJ</u> DATE <u>11/11/94</u> </div>		RSD: NMT 6.0%
<input checked="" type="checkbox"/> COMPLIES	PREPARED BY: <u>Nilesh Patel</u>	DATE: <u>11/11/94</u>
<input type="checkbox"/> DOES NOT COMPLY	APPROVED BY: <u>Suryakant Patel</u>	DATE: <u>11/11/94</u>

act13-147c

ide Pharmaceutical, Inc.

Page 2 of 2

LABORATORY TEST REPORT
FINISHED DRUG PRODUCT

PRODUCT: Digoxin Tablets, 0.5 mgSPECIFICATION: USPCONTROL #: 4300 ACHEMIST: KAVOLUME #: 326.01PAGE #: 144DATE: 11/1/94SAMPLE STAGE: Overall composite of the batch

TEST	RESULT	LIMIT
DISSOLUTION: Media: 500mL 0.1N HCl Appar: 1, rpm: 120 Temp: 37°C ± 0.5°C Time: 60 minutes	15 minutes: 1) <u>84.3</u> % 7) <u>82.9</u> % 2) <u>81.7</u> % 8) <u>82.0</u> % 3) <u>81.1</u> % 9) <u>82.2</u> % 4) <u>82.3</u> % 10) <u>78.8</u> % 5) <u>83.0</u> % 11) <u>79.7</u> % 6) <u>81.8</u> % 12) <u>79.9</u> % Average: <u>81.6</u> % 60 minutes: 1) <u>96.5</u> % 7) <u>90.0</u> % 2) <u>90.7</u> % 8) <u>89.1</u> % 3) <u>87.2</u> % 9) <u>88.3</u> % 4) <u>88.3</u> % 10) <u>88.3</u> % 5) <u>89.0</u> % 11) <u>86.5</u> % 6) <u>88.9</u> % 12) <u>88.5</u> % Average: <u>89.3</u> %	(Note - The specified tolerances are for % dissolved, and are not to be interpreted as Q values.) NLT 80% of the LC of Digoxin dissolved in 60 minutes for the average of 12 tablets tested and no individual tablet has less than 75% of the LC of Digoxin dissolved in 60 minutes. If the amount of Digoxin dissolved in 60 minutes is more than 95% for any individual Tablet, the amount dissolved in 15 minutes is not more than 90% for each individual Tablet. (LC: Labeled amount)
<div style="border: 1px solid black; padding: 5px; margin-top: 20px;"> APPROVED BY <u>SA</u> DATE <u>11/1/94</u> </div>		
<input checked="" type="checkbox"/> COMPLIES <input type="checkbox"/> DOES NOT COMPLY	PREPARED BY: <u>Nilesh Patel</u> APPROVED BY: <u>Suraj Patel</u>	DATE: <u>11/1/94</u> DATE: <u>11/1/94</u>

ac12-147d1

Amide Pharmaceutical, Inc.

Page 1 of 2

LABORATORY TEST REPORT**FINISHED DRUG PRODUCT**PRODUCT: Digoxin Tablets 0.5 mgSPECIFICATION: USPCONTROL #: 4301 ACHEMIST: P.K./NP/PA VOLUME #: 321-04/1306-2 PAGE #: 126/8/122 DATE: 11/9/94SAMPLE STAGE: overall composite up-batch (form fill up by PA)

TEST	RESULT	LIMIT
DESCRIPTION: Color:	<u>Green</u>	Green
Profile:	<u>Round bisected Tablet</u>	Round Bisected Tablets
Other: Debossed	<u>"A147" on bisected side of the tablet</u>	"A 147" on bisected side of the tablet
THICKNESS: (Guideline)	<u>3.4 mm</u>	3.0 mm to 4.0 mm
WEIGHT VARIATION:	<u>130.6 mg</u>	± 10% Theo. wt (130 mg) 117.0 mg - 143.0 mg
FRIABILITY:	<u>0.04 %</u>	NMT 1.0 %
IDENTIFICATION: (A)	<u>The retention time of the major peak in the chromatogram of assay preparation corresponds to standard preparation</u>	The retention time of the major peak in the chromatogram of Assay preparation corresponds to standard preparation.
ASSAY: Digoxin, 0.5 mg	<u>100.7 %</u>	90.0% to 105.0%
UNIFORMITY OF DOSAGE UNITS: (Content Uniformity)	1) <u>100.8 %</u> 6) <u>99.2 %</u> 2) <u>101.7 %</u> 7) <u>101.8 %</u> 3) <u>100.6 %</u> 8) <u>100.8 %</u> 4) <u>100.3 %</u> 9) <u>100.8 %</u> 5) <u>100.4 %</u> 10) <u>100.4 %</u> AV: <u>100.7 %</u> RSD: <u>0.7 %</u>	85.0% to 115.0%
() COMPLIES	PREPARED BY: <u>Nilesh Patel</u>	DATE: <u>11/1/94</u>
() DOES NOT COMPLY	APPROVED BY: <u>Suryakant Patel</u>	DATE: <u>11/1/94</u>

QC13-147c

de Pharmaceutical, Inc.

Page 2 of 2

LABORATORY TEST REPORT**FINISHED DRUG PRODUCT**PRODUCT: Digoxin Tablets, 0.5 mgSPECIFICATION: USPCONTROL #: 4301 ACHEMIST: PA VOLUME #: 33200 PAGE #: 122 DATE: 11/9/74SAMPLE STAGE: Composite sample (Form fill up by PA)

TEST	RESULT	LIMIT
DISSOLUTION: Media: 500mL 0.1N HCl Appar: I, rpm: 120 Temp: 37°C ± 0.5°C Time: 60 minutes	15 minutes: 1) <u>78.7</u> % 7) <u>80.6</u> % 2) <u>77.8</u> % 8) <u>77.7</u> % 3) <u>73.7</u> % 9) <u>78.7</u> % 4) <u>77.5</u> % 10) <u>77.0</u> % 5) <u>78.3</u> % 11) <u>75.7</u> % 6) <u>79.0</u> % 12) <u>77.4</u> % Average: <u>77.7</u> % 60 minutes: 1) <u>94.1</u> % 7) <u>90.7</u> % 2) <u>95.7</u> % 8) <u>91.0</u> % 3) <u>92.4</u> % 9) <u>91.7</u> % 4) <u>97.1</u> % 10) <u>90.2</u> % 5) <u>88.7</u> % 11) <u>89.0</u> % 6) <u>86.4</u> % 12) <u>87.3</u> % Average: <u>91.5</u> %	(Note - The specified tolerances are for % dissolved, and are not to be interpreted as Q values.) NLT 80% of the LC of Digoxin dissolved in 60 minutes for the average of 12 tablets tested and no individual tablet has less than 75% of the LC of Digoxin dissolved in 60 minutes. If the amount of Digoxin dissolved in 60 minutes is more than 95% for any individual Tablet, the amount dissolved in 15 minutes is not more than 90% for each individual Tablet. (LC: Labeled amount)
<input checked="" type="checkbox"/> COMPLIES <input type="checkbox"/> DOES NOT COMPLY	PREPARED BY: <u>Nilesh Patel</u> APPROVED BY: <u>Surajbhai Patel</u>	<div style="border: 1px solid black; padding: 5px; text-align: center;"> APPROVED BY <u>S.D.</u> DATE <u>11/11/74</u> </div> DATE: <u>11/11/74</u> DATE: <u>11/11/74</u>

qc12-167d1

AMIDE PHARMACEUTICAL, INC.

PROCESS VALIDATION PROTOCOL

DIGOXIN TABLETS 0.5 mg
MPR NO. 14702 REV. 00

BATCH SIZE: 400,000 TABLETS

PREPARED BY:

Admiral B. Q. Q.

Regulatory Affairs Director

DATE:

10/10/94

APPROVED BY:

Richard G. Nisala

Manufacturing Operations Director

DATE:

10-11-94

William E. Egan

Quality Assurance Director

DATE:

10/11/94

Surjajit Palit

Quality Control Director

DATE:

10/11/94

Richard G. Nisala

Vice President Operations

DATE:

10-11-94

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PROCESS VALIDATION PROTOCOL - DIGOXIN TABLETS 0.5 mg
MPR NO. 14702 REV.00

PURPOSE:

This document provides the procedure to be followed to validate the manufacturing process for Digoxin Tablets 0.5 mg. It applies to the next three consecutive batches to be produced.

SCOPE:

This protocol is designed to be prospective in nature.

The guidelines presented here include all steps of the manufacturing process which may have an impact on product quality. They are as follows:

Raw Materials
Blending
Compression

Details of the process will be found in the completed copies of the Manufacturing Batch Records which are available in the file. A summary of the process is found on the attached flow chart. The major equipment used will be documented and monitored as described in the appropriate section below.

Temperature and humidity will be monitored in the production area on a daily basis.

3% excess of Digoxin is added in the finished product to compensate for production losses.

The data gathered during the course of this study will be evaluated and any adjustments to the predetermined specifications or guidelines will be made as warranted based on the results of the three validation batches.

PROCEDURE:

RAW MATERIALS

All raw materials used in a validation batch will be certified to meet all current Amide specifications for that item. These will specifically include particle size profile, bulk density, and tamped density.

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Certification may be accomplished through direct testing by Amide, or an approved contract laboratory, or through a manufacturers Certificate of Analysis.

Digoxin, USP will be tested by Amide, or an approved contract laboratory for the complete monograph. This will include bulk density, tamped density, and particle size testing.

The excipients will be tested by Amide, or an approved contract laboratory, for those parameters required for expired stock retesting. In addition, particle size, bulk and tamped density will be run on all ingredients. The other results may be taken from the manufacturers COA.

In addition to the actual results, the name of the manufacturer, and the manufacturers lot number should be included in the report.

If more than one lot of a raw material is used in the production of the three batches the data should be evaluated to determine if any differences are detectable.

The acceptance criteria will be the specification limits for those tests listed in the Specification document.

BLENDING UNIFORMITY

The preblend will be produced in the 1 cu.ft. Twin Shell Blender, (#31). The speed will be monitored and documented both empty and during blending.

The blend in this step will be subjected to further processing, no sampling will be taken at this point.

The final blend will be produced in the 3 Cu Ft. Twin Shell Blender, (#32). The speed will be monitored and documented both empty and during blending.

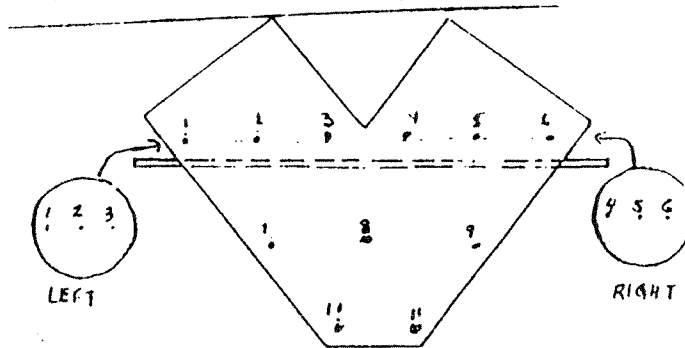
The sampling plan for the final blend is designed to evaluate overall blend uniformity, and those points in the blender where uniformity is most difficult to achieve. Samples are to be taken from the points shown below using only the 36 inch (small chamber) single port thief. The sample drawn should be about 390 mg which is three times the single dosage unit, and should be submitted to the laboratory in "Butter Paper."

AMIDE PHARMACEUTICAL, INC.

PROCESS VALIDATION PROTOCOL - DIGOXIN TABLETS 0.5 mg
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SAMPLING POINTS

- | | |
|------------------------------|--------------------|
| 1. Left Column - Top left | 7. Middle - Left |
| 2. Left Column - Top Center | 8. Middle - Center |
| 3. Left Column - Top Right | 9. Middle - Right |
| 4. Right Column - Top left | 10. Bottom - Left |
| 5. Right Column - Top Center | 11. Bottom - Right |
| 6. Right Column - Top Right | |



The samples are to be analyzed individually, without being ground, for Digoxin. No composite samples are to be prepared. The sample weight used for analysis should approximate 130 mg, which is the amount of this blend which would be present in one unit of the tablet.

Acceptance criteria is 85.0 - 115.0 % Th for the individual data points. This product has a 3% overage to compensate for the production losses.

One additional sample of about 150 g will be taken with the help of a stainless steel scoop from the top center of the blender. This sample will be tested for physical characterization which includes; bulk and tap density and particle size analysis. This data is for characterization only and these parameters will not be used to monitor routine production. Therefore, acceptance criteria will not be established.

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PROCESS VALIDATION PROTOCOL - DIGOXIN TABLETS 0.5 mg
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COMPRESSION

Compression will be accomplished using the stokes 45 station tablet press. The speed will be determined and documented during the validation study.

During compression samples will be collected every 15 minute by QA. These samples will be evaluated for individual tablet weight, thickness, and hardness. This will be 10 tablets for weight, and five each for thickness and hardness. Front and rear samples will be tested separately and will not be composited for any test in this section unless specifically stated.

The 15 minute samples should be arranged chronologically and the batch divided into thirds. Each third should be evaluated as described below for all tests except content uniformity. The samples for each test should be prepared by selecting, as close as possible, an equal number of tablets from each 15 minute sample. If selecting one tablet per 15 minute results in a greater number of tablets than the test requires the distribution should be as even as possible.

TEST	N
Friability	10 g - 1 Run
Disintegration	6
Dissolution	12 (6 front & 6 rear)

Content Uniformity testing is to be run across the entire batch. One tablet per 15 minute sample is to be run with a minimum of 30 tablets being required. The tablets selected for testing should be weighed prior to testing and their identity maintained. If compression runs for less than 8 hours, the additional tablets should be selected as evenly distributed as possible throughout the batch.

A portion of the blend will be run at hardness of 0.5 - 3.0 KP and above 8.0 KP. This will determine the effect of hardness on friability and dissolution.

Minimum quantities sufficient to equilibrate the press will be run at both lower and higher speeds. The actual ranges will be determined during production. Samples will be evaluated for hardness and weight.

Data analysis will consist of Average and Standard Deviation, with comparison both within and across the three batches. The data collected within each batch will also be evaluated for any possible trends.

AMIDE PHARMACEUTICAL, INC.

PROCESS VALIDATION PROTOCOL - DIGOXIN TABLETS 0.5 mg
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An overall composite sample will be prepared from all the 15 minute samples. This data will provide the basis for product release and will also be the initial data for stability.

Acceptance criteria will be as follows:

Target Weight (1 tablet):	130.0 mg
Target Weight (10 tablets):	1.300 g
Weight Range (1 tablets):	0.123 - 0.137 g
Thickness:	3.0 - 4.0 mm
Hardness:	2.0 - 8.0 KP
Friability	NMT 1%
Identification	Meets requirements.
Content Uniformity	85.0% - 115.0% (RSD NMT 6.0%)
Dissolution	Meets USP Requirement.
Assay	90.0 - 105.0%

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BATCH FLOW CHART FOR DIGOXIN TABLETS 0.5 mg
BATCH SIZE: 400,000 TABLETS
MPR # 14702, REV # 00

